

**A Dynamic Site Investigation
Adaptive Sampling and Analysis Program
for Operable Unit 1 at Hanscom Air Force Base
Bedford, Massachusetts**

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Abstract

In this study, field analytical instrumentation and methods were used to support a risk assessment study at the airfield at Hanscom Air Force Base (HAFB, Bedford, Massachusetts). The site investigation is part of an ongoing effort to determine contaminant risk to ground water from soil. The field tools supported a dynamic workplan/adaptive sampling and analysis demonstration. Over a two-week period, 601 soil samples were screened (30-sec/sample) for volatile organic compounds by direct measuring thermal desorption gas chromatography/mass spectrometry (TDGC/MS). From these results, 158 soil samples were selected for quantitative analysis by purge and trap GC/MS. In addition, quantitative analysis of 68 soil samples by TDGC/MS was made for polychlorinated biphenyls and polycyclic aromatic hydrocarbons. Quantitative analysis times were 10-min/sample for PCBs and PAHs and 15-min/sample for VOCs. A field-practical microwave digestion procedure and an inductively coupled plasma/optical emission spectrometry method were used to analyze 121 samples for metals. Results of the dynamic site investigation and field method performance are presented. Finally, HAFB staff modified the ground water collection system into the treatment plant increasing the influent concentration. This project was funded through the U.S. Environmental Protection Agency and the Environmental Technology Initiative.

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1.0 Introduction - Environmental Technology Initiative

In 1995, the U.S. Environmental Protection Agency (EPA) issued a Request for Proposals in support of President Clinton's efforts to promote innovative environmental technologies and to address the many factors that might pose barriers toward their commercialization. The President's Environmental Technology Initiative (ETI) is aimed at accelerating environmental protection, strengthening America's industrial base, and increasing exports of U.S. technologies and expertise. Through a cooperative agreement with EPA Region I and supported by ETI funding, Tufts University's Center for Field Analytical Studies and Technology (CFAST) addressed two key objectives identified in the FY95 strategic plan; namely, strengthening the capacity of technology developers and users to succeed in environmental innovation *and* strategically investing EPA funds in the development and commercialization of promising new environmental monitoring, control, and remediation technologies.

The U.S. Environmental Protection Agency *and* the Departments of Defense (DoD) and Energy (DoE) have sponsored programs to support research and to validate field analytical technologies.^{1,2} Despite EPA's efforts to encourage the use of field analytics,³ they have not played a significant role in either hazardous waste site investigations or the verification of hazardous waste site cleanup programs. The ETI project, in part, supported an ongoing soil investigation at Hanscom Air Force Base (HAFB, Bedford, MA). HAFB is in the process of conducting Human Health and Ecological Risk Assessments and Feasibility Studies for the airfield. The core technical team included staff from HAFB, EPA Region I, their respective contractors, CH2MHill and Camp Dresser & McKee (CDM), the Massachusetts Department of Environmental Protection (MA DEP) *and* Tufts University. The team developed and carried out a dynamic site investigation at Operable Unit 1. Dynamic site investigations depend on an adaptive sampling and analysis plan. The objective was to demonstrate the ability of field analytics to produce data of quality to support risk assessments. The HAFB investigation relied on data produced in the field to make decisions as to the location of samples collected and the types of analysis performed. Field instruments and methods were developed by the principal investigator (PI) in cooperation with several analytical instrument companies. The premise being that if analytical data can be produced in the field with known quality to support risk assessments then the perceived and/or institutional barriers impeding their usage should be greatly reduced.

With these objectives in mind, the technical team collaborated to produce an eighteen minute videotape illustrating the dynamic site investigation process.⁴ To complement the videotape, a dynamic workplan guideline was produced.⁵ The dynamic workplan provides a framework for changes in direction based on what is learned in the field during the site investigation or cleanup verification process. The guideline illustrates the many factors that should be considered when incorporating field analytical instrumentation and methods into an adaptive sampling and analysis program. The videotape and guideline are aimed at helping federal and state regulators, siteowners and their consulting engineers, and remediation companies understand what is involved in developing and carrying out a dynamic site investigation or cleanup verification program where the decisions made rely on field data. When compared with the traditional site characterization process, dynamic workplan/adaptive sampling and analysis projects should result in faster, better, and hopefully cheaper site investigations and cleanup.

Results of the HAFB soil investigation and the performance of the field instruments are presented. Direct measuring thermal desorption gas chromatography/mass spectrometry (TDGC/MS) was used to screen 601 soil samples for volatile organic compounds (VOCs). Based on these findings, 158 soil samples were quantitatively analyzed for VOCs by purge and trap GC/MS. Analysis times were 30-sec/sample and 15-min/sample for TDGC/MS screening and quantitative purge and trap GC/MS measurements, respectively.

Soil samples were selected for polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) analysis based on site history, the presence of petroleum contaminated soils observed in the field, and VOC screening results. Quantitative PCB and PAH analyses were made for 70 soil samples by TDGC/MS. Simultaneous analysis of these contaminants was accomplished in 10-minutes per sample. On-site analysis of 121 soil samples for the EPA target analyte list metals was made by a field-practical inductively coupled plasma/optical emission spectrometry (ICP/OES) method. Site contamination maps were produced to facilitate the on-site decision making process. The adaptive sampling and analysis program was completed in ten days. The data produced will be used in risk assessments to determine the need for future vadose zone soil actions. HAFB has already modified the ground water collection system. VOC influent concentrations into the treatment plant have increased from 500-ppb (August 1996) to 900-ppb (August 1997).

1.1 Hazardous Waste Site Investigation and Cleanup Costs

The EPA estimates that the cost for hazardous waste site cleanups will exceed \$300 billion over the next 10 years.⁶ In another study, Russel and coworkers⁷ project cleanup costs between \$480 billion and \$1 trillion over the next 30 years. These estimates exclude administration and transaction fees. Since 1980 the cost for Superfund alone has exceeded \$26 billion for both government and industry. More than 100 sites are now considered “cleaned” by the EPA, with another 275 sites expected to be in remedial design and construction by the year 2005.⁸ The U.S. General Accounting Office estimates that \$32 billion will be spent on these sites in long-term operation and maintenance monitoring costs through fiscal year 2040. Yet, another estimate suggests total remediation costs will range between \$44 billion (1,350 sites) to as high as \$138 billion (3,000 sites) for private non-Federal facility Superfund sites through the year 2020.⁹

The following questions can be posed: Do inadequate site investigations and, therefore, a lack of understanding with respect to the chemical and physical dynamics affecting the cleanup contribute to the costs? Can field-based analytical instrumentation and methods give on-site project engineers the kind of data needed that will lead to faster, better, and cheaper cleanups? Hanscom Air Force Base is an example where traditional site investigations have led to the construction of a ground water collection and treatment facility. Over the past 5-years, VOC concentrations into the plant have remained the same. These results have suggested the need for another field investigation aimed at determining whether plant operating conditions can be further optimized.

1.2 Hanscom Air Force Base Background

HAFB is in the towns of Bedford, Concord, Lexington, and Lincoln, Massachusetts. HAFB is approximately fourteen miles northwest of downtown Boston. From 1941 to 1973 HAFB's primary mission was the support of fighter aircraft operations and maintenance and the support of Air Force Research and Development (R&D). Thereafter, HAFB no longer provided fighter aircraft maintenance and began to support Air Force Command, Control, Communications, and Intelligence activities and R&D. Massachusetts obtained control of the airfield in 1974 and renamed it the L.G. Hanscom Field. The airfield is currently operated by the Massachusetts Port Authority as a civilian airport. Except for the airfield, the remainder of the base was retained by the Air Force.

HAFB and L.G. Hanscom Field were added to the National Priorities List in 1994. More than \$25 million has been spent on traditional hazardous waste site investigations and cleanups at Hanscom. Hazardous waste site investigations for Operational Unit 1 (Sites 1, 2, and 3) began at Hanscom Field in 1982, see Figure 1. Site 1 was used as a fire training area where waste oils, flammables, aircraft wreckage and fuselages were burned. Sites 2 and 3 are where fifty gallon drums containing waste solvents, fuels, and paints were buried. All visibly contaminated soils and drums were removed from these sites in 1987 and 1988.

In 1991, integrated ground water collection, recharge, and treatment systems were put into operation to remediate the three sites and to contain the plumes of contaminated ground water within the airfield. The ground water collection and recharge system and the 200-gal/min VOC treatment plant were built at a cost of \$6 million. Initial ground water influent concentrations were 10,000-ppb total VOCs. After six months of operation total VOC levels were 500-ppb. From late 1990 through July 1996 influent concentrations have remained constant, while yearly treatment facility operation and maintenance costs have increased to \$600,000/year.¹⁰

1.3 Traditional versus Dynamic Workplans

The ability to rapidly assess the disposition of environmental contaminants at purported or existing hazardous waste sites are an essential component of the nation's environmental restoration program. Each site, whether owned by the public or private sector, must be evaluated to decide whether risk to human health or the environment exists. If the data obtained supports the notion that either no risk or an acceptable level of risk exists for the intended land usage, then no further action may be required. If, on the other hand, sufficient risk has been determined to warrant a full site characterization, the site investigation effort must delineate the nature, extent, direction, concentration and rate of movement of the contamination along with the physical and chemical site attributes.

Sampling and analysis programs are a significant part of the environmental restoration process. From the initial site investigation on through to the design and completion of remedial actions, sampling and analysis programs play a key role. By design, traditional sampling programs collect soil or ground water samples based on pre-specified grid patterns in an attempt to maximize data while reducing chemical analysis costs, see Figure 2. They rely on

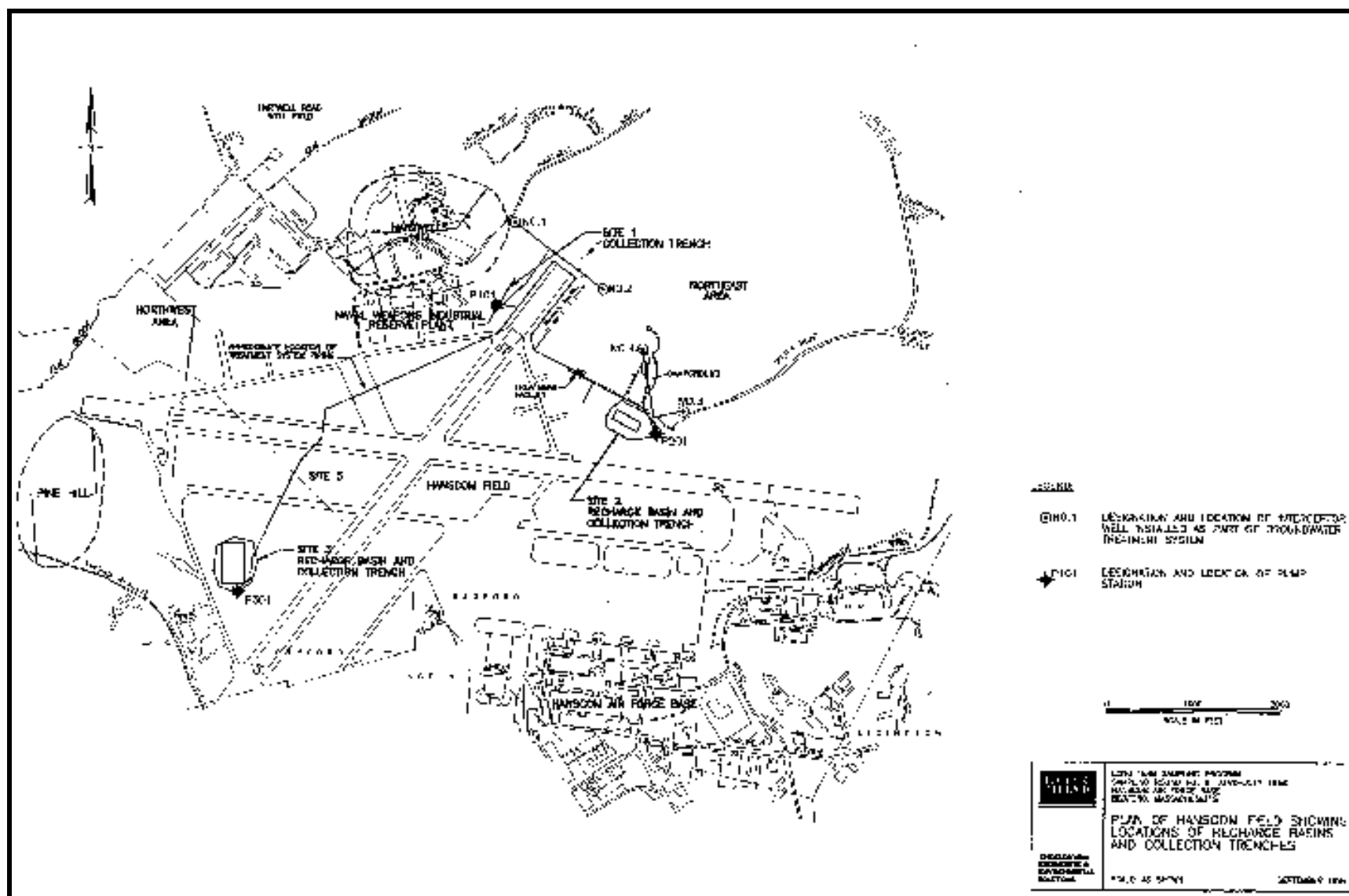


Figure 1. Hanscom Field

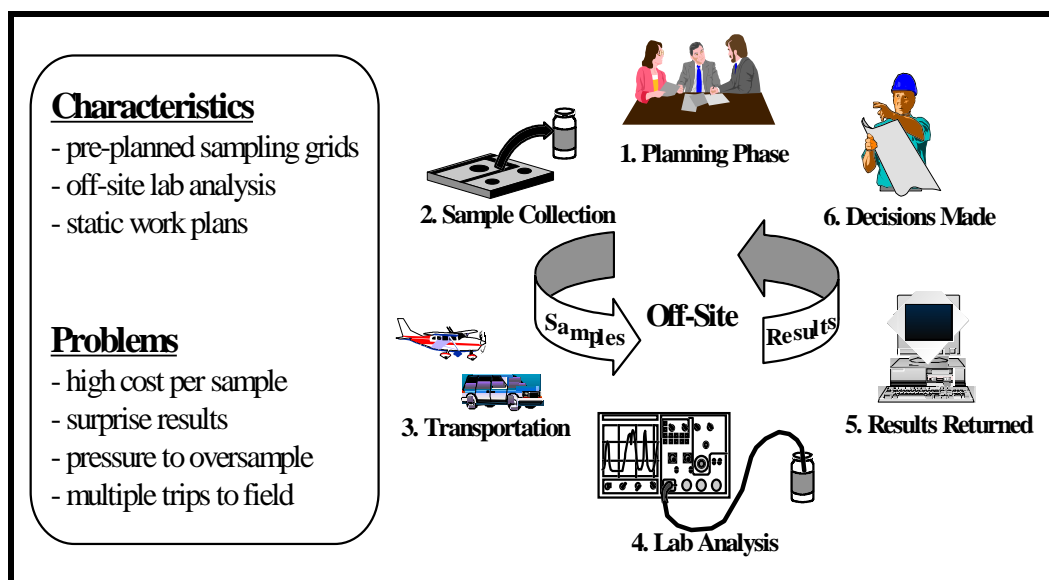


Figure 2. Traditional Site Investigation

predetermined specifications for the location and number of samples to be collected and the type of analyses to be conducted. Traditional site investigations are generally based on a phased engineering approach, which does not provide the framework for obtaining analytical data in the field nor for making changes in direction while in the field. Samples are collected, packaged and typically sent off-site for analysis. Because data turnaround times can range from a few days to several weeks, data “surprises” or concerns must be addressed in subsequent field studies. Each successive investigation continues to add cost to the overall restoration effort. Improvements in the way sampling and analysis programs are designed and executed should lead to faster, better, and more cost-effective site assessments and cleanups.

In contrast, adaptive sampling and analysis programs are based on a dynamic workplan where the program itself relies on field analytical instrumentation and methods to generate near real-time information on the nature, extent, direction, concentration and rate of movement of the contamination present at the site. Figure 3 illustrates the dynamic nature of this approach. Rather than dictate the details of the sample analyses to be performed, the numbers of samples to be collected, and the location of each sample; dynamic workplans specify the decision making logic that will be used in the field to determine what analyses will be performed, where the samples will be collected, and when sampling stops. Adaptive sampling and analysis programs change or adapt based on the analytical results produced in the field.

Successful hazardous waste site investigations should be focused with goals and objectives clearly defined. A dynamic workplan provides an alternative to the traditional approach. It relies, in part, on an adaptive sampling and analysis strategy.¹¹ An adaptive sampling and analysis program requires analytical methods and instrumentation that are field-practical and can produce data fast enough to support the dynamic workplan process. Past studies have shown that the dynamic site investigation process employing field analytics can result in significant time and cost savings.^{12,13} A properly developed dynamic workplan includes the following six steps summarized below:⁵

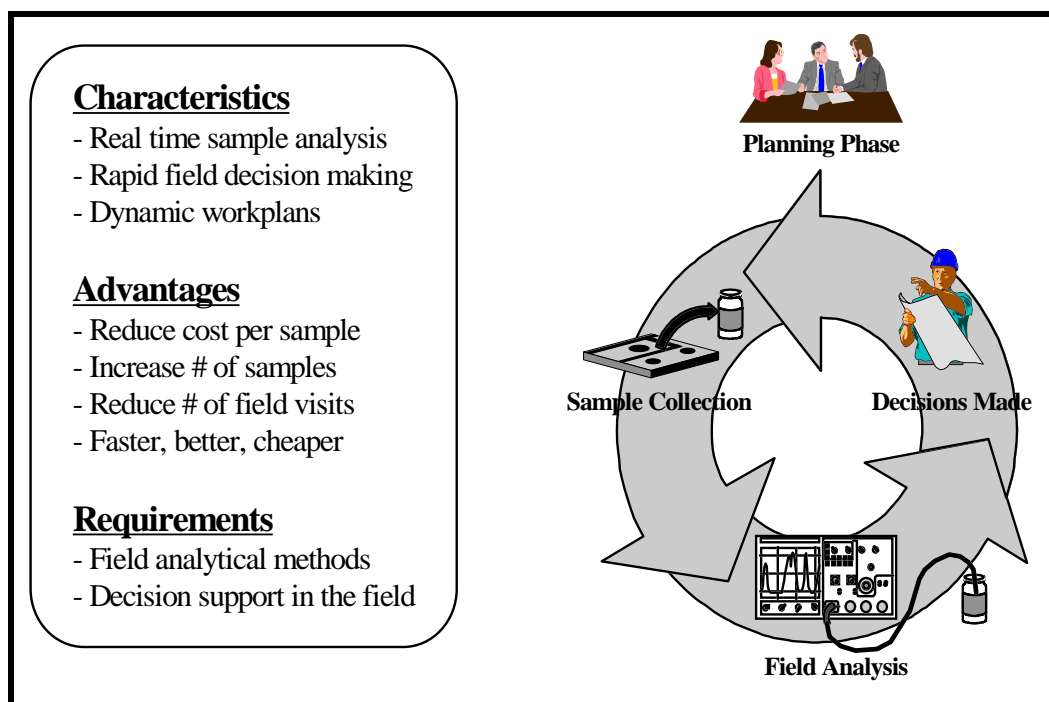


Figure 3. Adaptive Sampling and Analysis Program

- Step 1: Select the core technical team whose responsibility it will be to prepare and carry out the dynamic workplan. The core technical team must understand the scientific and engineering questions under investigation and the quality of data needed to answer these questions. One member of the team should have the authority to make field decisions.
- Step 2: Develop the Initial Conceptual Model and Decision Making Framework. The model contains the best-available information at the start of the project and evolves as field data is produced. It depicts three-dimensional site profiles based on vadose zone and ground water flow systems that can exert influence on contaminant movement. The model is based on site-specific Data Quality Objectives (DQO's). DQO's ensure that the type, quantity, and quality of field data used in decision making are appropriate for the intended application.
- Step 3: Develop Standard Operating Procedures. SOPs for sample collection and analysis should be produced by the core technical team and approved by the appropriate regulatory body before initiating field activities. The field methods should be "performance based" and provide data of sufficient quality to achieve site-specific DQO's, with sample analysis rates that can support the dynamic site investigation process.
- Step 4: Develop Data Management Plan. The ability to manage and easily use all of the data (chemical, physical, geological, hydrological) produced in the field is critical to the success of the dynamic process. Data integration, sampling, and analysis protocols should be incorporated into an overall data management plan.

- Step 5: Develop Quality Assurance Project Plan. Quality assurance/quality control (QA/QC) defines the responsibility of the technical team and regulators. It describes the procedures to be used to monitor conformance with, or documentation and justification of departure from the SOPs.
- Step 6: Prepare Health and Safety Plan. A health and safety plan is produced with DQO's established for the field analytical tools used to monitor worker and community safety.

2.0 Site Investigation Purpose and Objectives

HAFB is completing its Human Health and Ecological Risk Assessments and Feasibility Study for Operable Unit 1. The effectiveness of the 1987/88 drum and soil removal actions and five-year operation of the ground water collection, recharge and treatment systems must be assessed. The core technical team's primary objective was to show that the field analytical instrumentation and methods can support a dynamic workplan/adaptive sampling and analysis program. The quality of data produced in the field will support its use in the risk analysis *and* in assessing the effectiveness of past removal actions. Another goal was to videotape the development of the workplan and execution of the site investigation. A third goal was to produce a dynamic workplan/field analytics guideline. The videotape and guideline should provide a framework for making decisions in the field and assist in technology transfer. Finally, interviews were conducted with technology developers and manufacturers, field instrumentation purchasers, and data users to identify barriers that pose impediments to the use of field analytics.¹⁴

3.0 Methodology

When the ETI proposal was submitted for funding, several facilities were identified as potential locations for the dynamic site investigation program. The premise being that an ideal site is one in which the soil investigation program was in the planning stage. This would allow ETI funds to be used to demonstrate field analytics and the dynamic investigation process. At the time ETI funding was received, HAFB was the best available site in Region I.

Just before mobilization the core technical team held its final field investigation planning session at Tufts University. Details of the site investigation objectives, sample collection process, field analyses to be performed, and the framework for making decisions in the field were finalized.¹⁵ The planning session was videotaped, with staff from each organization immediately interviewed after the meeting to gain perspective. Creative Video (Medfield, MA) developed the interview questions and videotape format with the assistance of the PI. HAFB staff assumed primary responsibility for directing the sample collection effort. When questions were raised concerning measured contaminant concentrations at the action level, EPA provided guidance to determine whether additional sampling was required. The work performed was conducted under an EPA approved workplan. The Agency conducted laboratory audits, reviewed SOP's and method detection limit (MDL) studies, *and* verified the data. Staff from Tufts and CH2MHill prepared chain-of-custody forms and logged information about the samples. Tufts prepared

samples for field and off-site laboratory analysis, while CH2MHill was responsible for shipping samples to the off-site laboratory. Field analysis for organics and metals was provided by Tufts, while Spectrum Analytical (Agawam, MA) performed the off-site laboratory analysis. Soil samples were collected in 4-ft plastic tubes using a Geoprobe™ operated by Kestral (Agawam, MA). Project milestones including site selection, dynamic workplan preparation, field mobilization and investigation concomitant with videotape production, data and final report submission are shown in Appendix II.

3.1 Adaptive Sampling and Analysis Strategy

The site-specific action levels (i.e. 20DAF), quantitation limits (QL), and method detection limits (MDL) for the compounds of interest are shown in Table 1. 20DAF is derived from EPA's Soil Screening Levels (SSL)¹⁶ and was established as the action level for determining risk to ground water. 20DAF is the dilution-attenuation factor (DAF) of 20, which takes into account the natural attenuation processes for the soil to ground water migration pathway. For Operable Unit 1, one-half the action level (i.e. $\frac{1}{2} \times 20\text{DAF} = 10\text{DAF}$) was established as the measurement quantitation limit to insure that site-specific action concentrations were achieved. For convenience, the 10DAF values were used to produce the site maps.

Adaptive sampling and analysis programs require that on-site chemical analyses be fast enough to support the sample collection and decision making process. For Sites 1, 2, and 3, VOCs, PCBs, PAHs, cadmium, and lead were the contaminants of concern. Field instruments, their corresponding rate of analysis, SW 846 reference method, and analysis type are shown below for each field method used in this investigation.

<u>Field Methods</u>	<u>Sample Introduction and Instruments</u>	<u>Analysis Type</u>	<u>Sample Analysis Rates</u>
Metals Modified SW 846 Method 6010	Hildebrand nebulizer Leeman ICP/OES	Quantitative	8-min
PCBs and PAHs Modified SW 846 Method 8270B	Tufts TD & Hewlett Packard GC/MS	Quantitative	10-min
VOCs Modified SW 846 Method 8260A	Tekmar purge and trap & Hewlett Packard GC/MS	Quantitative	15-min
VOCs Tufts method	Bruker TD & Bruker GC/MS	Screening	< 30-sec

Table 1. Site-specific Action Levels, Quantitation Limits, and Method Detection Limits

Compound	Action Level	QL	MDL
	20DAF (mg/kg)	10DAF (mg/kg)	
Acenaphthene	570	285	0.1
Acenaphthylene	570	285	0.1
¹ Anthracene	12,000	6,000	0.2
Benzene	0.03	0.015	0.003
² Benz(a)anthracene	2	1	0.2
³ Benzo(b)fluoranthene	5	2.5	0.3
³ Benzo(k)fluoranthene	490	245	0.3
Benzo(a)pyrene	8	4	0.1
Cadmium	8	4	0.11
Carbon Tetrachloride	0.07	0.035	0.004
Chlorobenzene	1	0.5	0.008
Chloroform	0.6	0.3	0.008
² Chrysene	1600	800	0.2
Dibenz(a,h)anthracene	2	1	0.2
1,1-Dichloroethane	23	11.5	0.006
1,2-Dichloroethane	0.02	0.01	0.013
1,1-Dichloroethene	0.06	0.03	0.003
cis-1,2-Dichloroethene	0.4	0.2	0.005
trans-1,2-Dichloroethene	0.7	0.35	0.006
Ethylbenzene	13	6.5	0.006
Fluoranthene	43,000	21,500	0.1
Indeno(1,2,3-cd)pyrene	14	7	0.2
Lead	400	200	1.65
Naphthalene	84	42	0.4
¹ Phenanthrene	NA	280	0.2
Pyrene	42,000	21,000	0.1
Total PCBs	NA	0.5	0.2
Styrene	4	2	0.006
Tetrachloroethene	0.06	0.03	0.006
Toluene	12	6	0.010
1,1,1-Trichloroethane	2	1	0.008
Vinyl Chloride	0.01	0.005	0.033
⁴ m-Xylene	210	105	0.016
o-Xylene	190	95	0.003
⁴ p-Xylene	200	100	0.016

Notes: Organics with the same superscript co-elute. EPA has not established a 20DAF for total PCBs, therefore, the site-specific quantitation limit was set as 0.5-mg/kg. No 20DAF concentration was available for lead. One-half of the screening level of 400-mg/kg for ingestion was used based on the Revised Interim Soil Lead Guidance for CERCLA Sites and RCRA Corrective Action Facilities (U.S. EPA 1994).

Geoprobe™ push technology was used to collect subsurface soils in 4-ft plastic sleeves. VOC screening analysis was made at 1-ft intervals by making a small incision along the sleeve, with the TDGC/MS sampling probe head placed immediately over the hole. Volatile vapors present in the soil were instantaneously detected by the MS, which was operated in the continuous direct measuring mode. The Bruker TDGC/MS simultaneously monitored eleven of the eighteen targeted VOCs . To insure compound identity, three ions per compound were monitored. Soil samples were collected by rounds at each site as follows:

- Round 1- Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water at the center of each fire training and drum burial pit.
- Round 2 - Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water outside the fire training and drum burial pits. Soil samples were also collected near the upper aquifer collection trenches to assess contamination migration toward the trenches.
- Round 3 - Geoprobe™ sampling was performed in continuous 4-ft increments from the surface to ground water further out from the conceptualized fire training and drum burial pit boundary whenever GC/MS screening and quantitative data from Round 2 produced detectable levels above the 10DAF quantitation limit. Note that the site-specific action level for all three sites was set at 20DAF.

Rounds 1 and 2 were prespecified in the dynamic workplan and collected as a group from each site. Both screening and quantitative data dictated whether additional samples from Rounds 1, 2, or 3 required further analysis, for example:

- If GC/MS screening results indicated non-detectable VOC levels within the 4-ft sleeve for all sleeves from a particular boring, a soil sample for quantitative GC/MS analysis was selected from the 2-ft section of the sleeve nearest to ground water.
- If only one 4-ft sleeve from a boring produced screening level concentration at detectable levels, a soil sample was selected for quantitative analysis from the 2-ft section of the sleeve within the area of highest concentration. An additional soil sample was selected for quantitative analysis from the 2-ft section of the sleeve nearest ground water whenever the sample selected by screening was not from the sleeve nearest the ground water level.
- If target compounds were present in multiple 4-ft sleeves within the same boring above ground water, a soil sample was selected for quantitative analysis from the 2-ft section of the sleeve shown to be the area of highest concentration. Additional soil samples were selected for quantitative

analysis from the 2-ft section of the sleeve nearest ground water whenever the sample selected by screening was not from the sleeve nearest the ground water level. Supplementary samples were selected for quantitative analysis to determine extent of contamination from these boring locations.

3.2 Field Instruments, Sample Preparation and Analysis Procedures

All soil samples were prepared and analyzed in the field. Detailed descriptions of the sample preparation methods, analyte quantitation procedures, and quality control and assurance criteria can be found in the VOC, semi-VOC, and metals standard operating procedures (SOPs), see Appendix III. Data reduction for quantitative VOC and PCB/PAH GC/MS analysis was accomplished in the data management trailer. Off-line data analysis maximized the sample throughput rates of the quantitative methods. Screening results were instantaneous, with instrument response visible on the monitor and recorded manually. Data reduction for metals was made in the metals trailer using the Leeman Laboratory data analysis software. Several software mapping programs were proposed as part of the ETI project. Once enough organics and metals data were generated, site maps were produced using SitePlanner™ (Consolve, MA) to visualize site contamination profiles. Daily inspection of the maps (versus tables of data) facilitated the on-site decision making process. Plume™ (a geostatistical sampling tool) was not used in the investigation. Site dimensions at each site in Operable Unit 1 were too small to use the program.

3.2.1 Organics Analysis

A Bruker Instruments (Billerica, MA) GC/MS was used to provide direct measuring screening data of VOCs in soil. Each 4-ft sleeve was marked at 1-ft intervals along the length of the tube as described earlier. A hole was cut at the center of each 1-ft section. The 3-ft TD sampling probe head was held directly over the hole and the signal response for each target analyte recorded.¹⁷ If no response was noted after 1-min the section was considered blank. This procedure was repeated for each 1-ft interval of the sleeve, where possible. The data generated using this screening method was used to determine which, if any, 2-ft section was to be sampled for quantitative GC/MS analysis.

Two different VOC purge and trap devices were used in the ETI study. The Tekmar 3000 sample introduction system was used with a Hewlett Packard (HP, Palo Alto, CA) GC/MS to provide quantitative analysis of all soils in the HAFB soil screening investigation/adaptive sampling and analysis program. The quantitative VOC measurements were used to confirm the screening data, while both screening and quantitative data were used to delineate the extent of contamination. The Tufts VOC purge and trap/thermal desorption (TD) GC sample introduction system, designed as a lower cost alternative (\$5,000) to the Tekmar (\$15,000) for field application, was initially intended to be used in the field investigation. Because of carrier gas line leakage and the start of the sampling program, the Tekmar sample introduction system became the primary means of producing VOC data. Purge and trap GC/MS is the standard laboratory method for quantitative VOC analysis. Sample introduction by Tekmar provided a

common baseline when comparisons were made between the HP GC/MS data analysis software (EnviroQuant) and the mathematical algorithms (Ion Fingerprint Detection™ software) developed at Tufts. The HP model 5890 II/5972 GC/MS was used with the Tekmar. The HP GC/MS model GCD was used with the Tufts purge and trap system. The field method for VOCs was modified from the EPA standardized SW 846 method 8260A, see Appendix III.

Unlike the Bruker TD, the Tufts thermal desorber was not used to measure organics from soil directly. For PCB (by homologs) and PAH (compound-specific) analysis, 2-g soil was extracted with 2-ml methylene chloride, with known aliquots (50-ul) of the extract injected into a disposable glass sleeve in the TD. The thermal desorber was installed into the GC injection port of the HP GC/MS model GCD. Standard syringe injection techniques (typically 1 to 2-ul) cannot provide the detection limits achieved by the TD without preconcentrating the soil extract. Extensive sample cleanup and/or fractionation may also be needed depending on the complexity of matrix interferences. PCBs and PAHs were measured in the same 10-min analysis. The PCB/PAH field method was modified from previously published studies using the Bruker TDGC/MS^{18,19,20,21} and from reference method SW 846, 8270B, see Appendix III. In this project, HP's mass spectrometry detection system and data acquisition software were used to provide quantitative data.

All data analysis software comparisons were made using the same MS data files, with either Tufts' IFD or HP's EnviroQuant software. All computer systems were linked to a central data processing work station in the data management trailer through a local area network. All of the GC/MS data was backed up on removable storage media (Iomega Zip100 drives).

3.2.2 Metals Analysis

A Leeman Laboratory (Lowell, MA) model PS-1000 ICP/OES was modified to provide on-site analysis of metals. Typical ICP/OES instruments require highly temperature controlled laboratory environments, $\pm 5^{\circ}\text{C}$, or optical components drift quickly out of calibration. This temperature requirement is costly to achieve in field laboratories especially during the extreme summer and winter months. The Leeman ICP/OES is a scanning sequential spectrometer. Modifications were made by pneumatically locking in place the optical system's movable components including the optical plate and Photo Multiplier (PMT) sled. The chassis was also ruggedized to increase instrument stability during transport and field operation. These design changes from traditional laboratory instruments to a more field-practical instrument were made by the Leeman and Tufts staff. We also modified the standard ICP/OES spray chamber with a Hildebrand grid nebulizer. This system can handle samples whose total dissolved solids content is greater than 1,000-ppm and digestate acid concentrations greater than 25% by volume. The spectrometer and Argon plasma torch were powered in the field by two different electrical services, 110-V 20-Amp and 240-V 30-Amp. The field ICP/OES instrument has been used at site investigation projects conducted with the Departments of Defense²² and Energy.²³

The ICP/OES was calibrated by a standard ratio method between the signals from blank and standard solutions. A laboratory control standard, prepared from an ERA Waste WatR™ solution, was diluted to approximately the instrument limit of quantitation. Analysis of this standard was used to verify instrument response. Instrumental QA/QC solutions consisted of

calibration blanks, calibration verification check standards, interferant A/B solutions, and laboratory control solutions. Instrument standards were purchased from Leeman, laboratory control standards from ERA (Arvada, CO) and Plasma-Chem (Farmingdale, NJ). Calibration responses were made for each metal and then verified with check standards. After the analysis of ten samples, instrument stability was checked by analyzing a check standard and performing a continuing calibration verification check with the concentration falling within a $\pm 20\%$ range. Calibration blanks were run before calibration verification standards. Interferant solutions A and B were run before site samples were analyzed. Details of the Tufts microwave digestion sample preparation procedure and ICP/OES analysis can be found in Appendix III.

Metals data was also produced from an energy dispersive x-ray fluorescence (EDXRF) instrument. The Spectrace QuanX differs from other field XRF instruments by employing an x-ray tube as the source rather than multiple radionuclides such as ^{55}Fe , ^{109}Cd , and ^{241}Am . It is tabletop in size and employs a thermoelectrically cooled Li drifted silicon semiconductor detector as opposed to liquid nitrogen cooled detectors typically found in laboratory instruments. This instrument provides increased sensitivity over radionuclide source instruments and has the same sensitivity as the much larger x-ray tube-based XRF's.

Soil samples were placed into a 32-mm mylar windowed sample cell. Samples were analyzed for 120 livetime seconds each for lead and cadmium. Fundamental parameter calculations were made to determine lead and cadmium concentrations in addition to producing the Pb ($L\beta$) and Cd ($K\alpha$) emission signal versus concentration plots based on standard reference materials (SRM) 2704, 2709, and 2710 obtained from National Institute of Standards and Testing. HAFB soil samples were analyzed with the field XRF after the completion of the soil screening investigation. The data from this instrument was produced as part of the ETI technology transfer project to determine instrument performance, see SOP in Appendix III for method details.

3.2.3 Quality Assurance/Quality Control Procedures

As was established in the dynamic workplan/adaptive sampling and analysis strategy, the HAFB site-specific data quality objectives were established to provide data of sufficient quality to support a risk analysis and the effectiveness of prior removal actions. Tables 2 and 3 list the data quality objectives for quantitative analysis of organics and metals.

3.2.4 Qualitative Identification and Quantitation Methods

Organics The Bruker mass spectrometer was operated in the selected ion monitoring mode to provide rapid detection of VOCs. Compound identity was made when the selected ions (three per compound) were normalized to 100% at the peak maxima and on either side at the half-peak maxima on three consecutive scans through the chromatographic peak. Signal amount, i.e., the logarithmic value of the SIM signal, was recorded when the above criteria was met. Concentrations were not calculated, relative signal response was used to guide sample location selection and the selection of samples analyzed quantitatively in the field.

Table 2. HAFB Data Quality Objectives for Quantitative Analysis of Organics

<u>Initial Calibration:</u> 5-point calibration	<u>Requirements</u> ave RF %RSD +/- 30% for 2/3 and +/- 40% for remaining 1/3 target compounds	<u>Corrective Action</u> check standards, recalibrate, check instrument
<u>Continuing RF Calibration:</u> begin and end of day	+/- 30% difference between ave RF and daily RF (CCV) for 2/3 and +/- 40% difference for remaining 1/3 of target compounds	check standards, recalibrate, check instrument
<u>Method Blank:</u> beginning and end of day or after analysis of highly contaminated sample	No more than 4 target compounds, concentration < 3 times QL	use new source of deionized water
<u>Measurement Precision:</u> duplicate or triplicates every 20 th sample	concentrations > 5 x QL: RPD < 60% concentrations < 5 x QL: RPD < 100%	review lab QC to determine whether in control, if out of control flag data
<u>Measurement Accuracy:</u> 1) surrogate fortified samples 2) field versus laboratory comparison	1) between 30% and 200% 2) concentrations > 5 x QL: RPD < 60% concentrations < 5 x QL: RPD < 100%	1) flag data 2) not applicable

The HP mass spectrometers were operated in the total ion current (TIC) mode for both VOC and semivolatile analyses. The software (IFD) extracted selected ions and their intensity for each compound. It utilized a set of mathematical algorithms and predetermined retention windows to identify and quantify each compound. The retention windows were determined from the 5-point linear calibration separation for each compound. Positive identification required the detection of the primary ion and at least two secondary ions for each target compound. Target compound quantitation was based on the integrated abundance of the primary ion and was calculated as follows:

$$\text{Concentration (ng/g)} = K(A_x)(C_{is})/(A_{is})(RF)(W_s)(D)$$

where, A_x = integrated area of primary ion, C_{is} = concentration of internal standard, A_{is} = integrated area of internal standard, $RF = (A_{std}) (C_{is}) / (A_{is}) (C_{std})$, K = dilution factor, W_s = weight of the sample (g), and $D = (100 - \% \text{moisture in the sample}) / 100$. The average response factor, RF, for each compound was determined over the 5-point linear calibration curve.

Table 3. HAFB Data Quality Objectives for Metal Analytes

<u>Initial Calibration:</u> 2-point calibration, a blank and one known high level concentration	<u>Requirement</u> average of three solutions	<u>Corrective Action</u> check standards, recalibrate, check instrument
<u>Continuing Calibration Verification:</u> every 10 th sample	percent recovery +/- 20%	check standards, recalibrate, check instrument
<u>Instrument Blank:</u> every 10 th sample	concentration below reporting limit	scan selected wavelengths, increase rinse time, reanalyze acid solution
<u>Method Blank:</u> every 20 th sample	concentration below reporting limit	scan selected wavelengths, increase rinse time, prepare new samples and reanalyze
<u>Measurement Precision:</u> duplicate every 20 th sample	+/- 25% RPD	review lab QC, determine whether in control, if in control flag data
<u>Measurement Accuracy:</u> 1) laboratory control check samples (ERA soil and solution) analyzed every 20 th sample 2) field versus laboratory	percent recovery +/- 20% a) +/- 60% RPD and b) $50\% < R < 200\%$ where $R = 100 \times C_{\text{on-site}}/C_{\text{off-site}}$	1) review lab QC, determine whether in control, if in control flag data 2) site-specific

Metals Metals were identified by their characteristic atomic emission wavelengths. Each metal has a unique wavelength at which spectral interferences were minimal. The area count was obtained by integrating the plot of emission intensity versus wavelength. The metal was quantified by calculating the concentration from a 2-point calibration plot (matrix blank and one known high level concentration) as follows:

$$\text{Concentration (ng/g)} = (C_s)(A_x)/(A_s)$$

where C_s = concentration of calibration standard, A_x = sample area count, and A_s = calibration standard area count.

4.0 Results and Discussion

The quality of data produced in the dynamic site investigation will support its use in the HAFB risk assessment *and* in deciding if additional actions are needed in the vadose zone soil. Described below are the results of the adaptive sampling and analysis program *and* the performance of the field instruments and methods.

4.1 Adaptive Sampling and Analysis Results

The lowest calibration standard from the 5-point curve established the quantitative GC/MS reporting limits (RL). The RL's were supported by method detection limit studies for the targeted organics and metals of concern. Table 1 lists the action level, the corresponding quantitation limit, and MDL for each analyte, see Sections 4.2.2 and 4.2.3 for results. Site contamination maps are expressed at each target compounds' 10DAF concentration (the QL) for ease of visual inspection, see Figure 4 for map key.

Figures 5 and 6 depict surface to bedrock cross sectional profiles for Sites 1, 2, and 3.^{15,24} The maps also show the initial conceptual model for each site used to help guide the sample collection process and locate wells found to contain high levels of VOCs. The depths to critical horizons below the ground surface were:

- depth from surface to pit bottoms (3-ft Site 1, 6-ft Sites 2 and 3),
- pit bottoms to top of recharge basin (~ 12-ft Sites 2 and 3),
- depth from surface to top of lacustrine unit (Site 1 not available, Site 2 ~ 15-ft, Site 3 ~ 18-ft),
- depth from surface to bottom lacustrine unit (Site 2 21-23-ft, Site 3 23-ft to 26-ft),
- depth from surface to bedrock (Site 1 15-20-ft, Site 2 40-ft, Site 3, 90-ft),
- depth to bedrock from top of recharge basin (Site 1 not applicable, Site 2 46-ft, Site 3 95-ft)

An average of 75 soil samples was screened for VOCs per day by TDGC/MS over a two-week period for a total of 601 samples analyzed. Quantitative VOC analysis of 158 soil samples by purge and trap GC/MS was made to confirm the screening results and to delineate the extent of contamination. Quantitative analyses of 70 soil samples for PCBs and PAHs and 121 soil samples for metals were made. Described below are our findings for Sites 1-3 *and* the collection and analysis decisions made in the field.

Icons of Samples:

△ Qualitative ND

The sample comes from a boring that was ND for all depths during screening and confirmed by one quantitative analysis for the boring. For example in [Site 2](#) the samples relating to sample location [S2-B02-1A](#) are [light blue](#) for samples [above 8 feet](#), and [dark blue](#) for samples [below 8 feet](#). This means that each boring contained non detectable levels of target compounds as analyzed by qualitative fast-GC/MS. Confirmation analysis by quantitative GC/MS and Tufts data interpretation software for one sample showed the same result. In this case the confirmation sample was from a depth below 8 feet.

□ Quantitative, concentration less than 10DAF

The sample was quantitated using GC/MS and Tufts data interpretation software and was either an ND or was found to contain contaminants in lower concentration than 10DAF.

○ Quantitative, concentration more than 10DAF

The sample was quantitated using GC/MS and Tufts data interpretation software and was found to contain contaminants at higher concentration than 10DAF.

Note: To normalize* the action levels, all target compound concentrations have been divided by their corresponding 10DAF value ($\equiv 1/2$ of the 20DAF value). The maps indicate contaminants above the 10DAF.

* Calculation example, Site 2 - Chlorinated VOCs below 8ft, sample S2-B13-(14-16):
380 ppb cis-1,2-dichloroethene, 10DAF = 200 pbb, $380/200 = 1.9$ -10DAF
42 ppb tetrachloroethene, 10DAF = 30 ppb, $42/30 = 1.4$ -10DAF

Compound numbering and DAF values:

#	Compound	10DAF
1	Vinyl Chloride	5 ppb
2	Methylene Chloride	10 ppb
3	1,1-Dichloroethene	30 ppb
4	1,1-Dichloroethane	10 ppb
5	cis-1,2-Dichloroethene	200 ppb
6	trans-1,2-Dichloroethene	300 ppb
7	Chloroform	300 ppb
8	1,2-Dichloroethane	10 ppb
9	1,1,1-Trichloroethane	1000 ppb
10	Carbon tetrachloride	35 ppb
11	Trichloroethene	30 ppb
12	Benzene	15 ppb
13	Tetrachloroethene	30 ppb
14	Toluene	6000 ppb
15	Chlorobenzene	500 ppb
16	Ethylbenzene	6500 ppb
17	Styrene	2000 ppb
18	o-Xylene	95000 ppb
19	m/p-Xylene	105000 ppb

Figure 4. Map Key

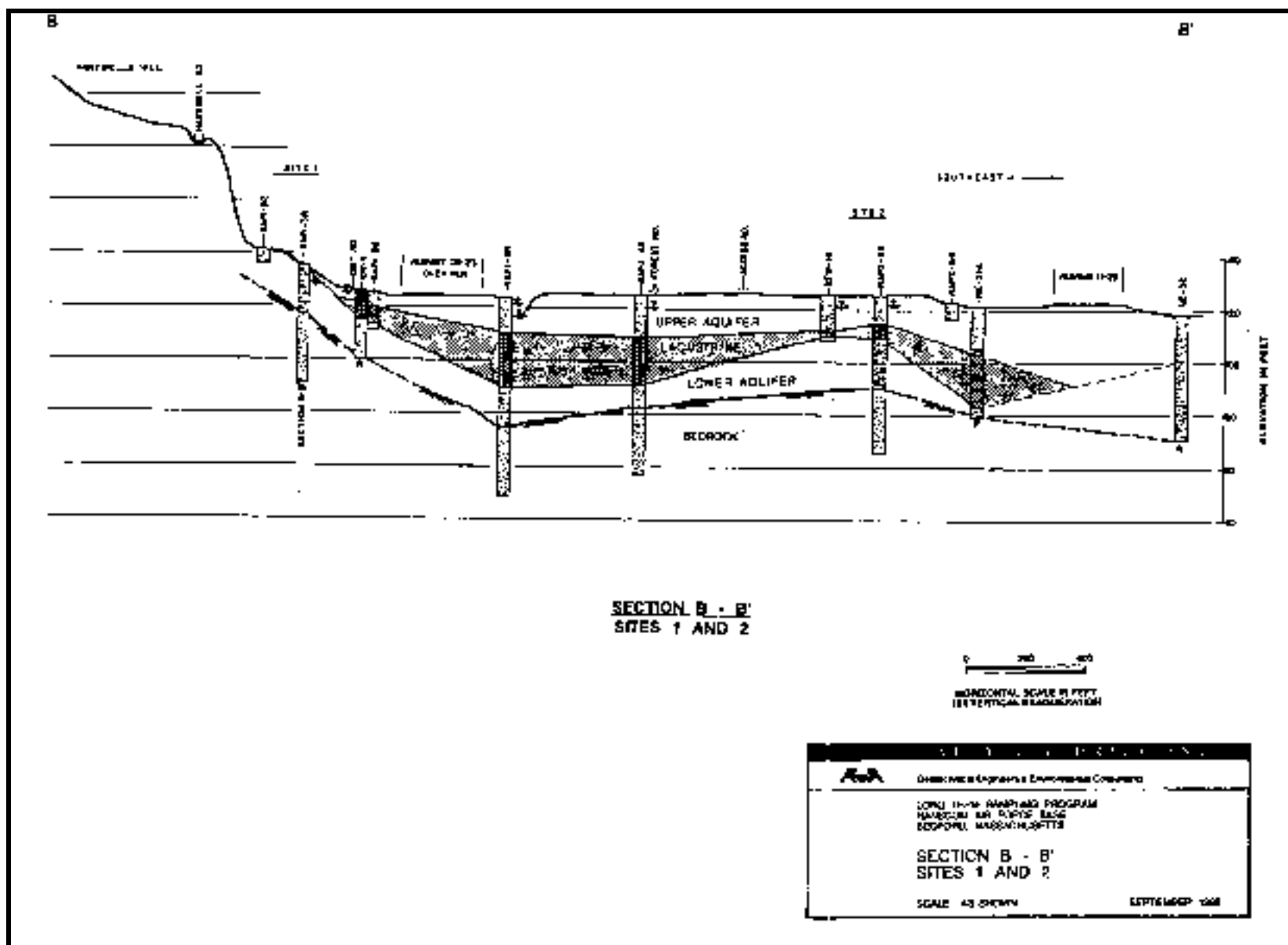


Figure 5. HAFB Sites 1 and 2

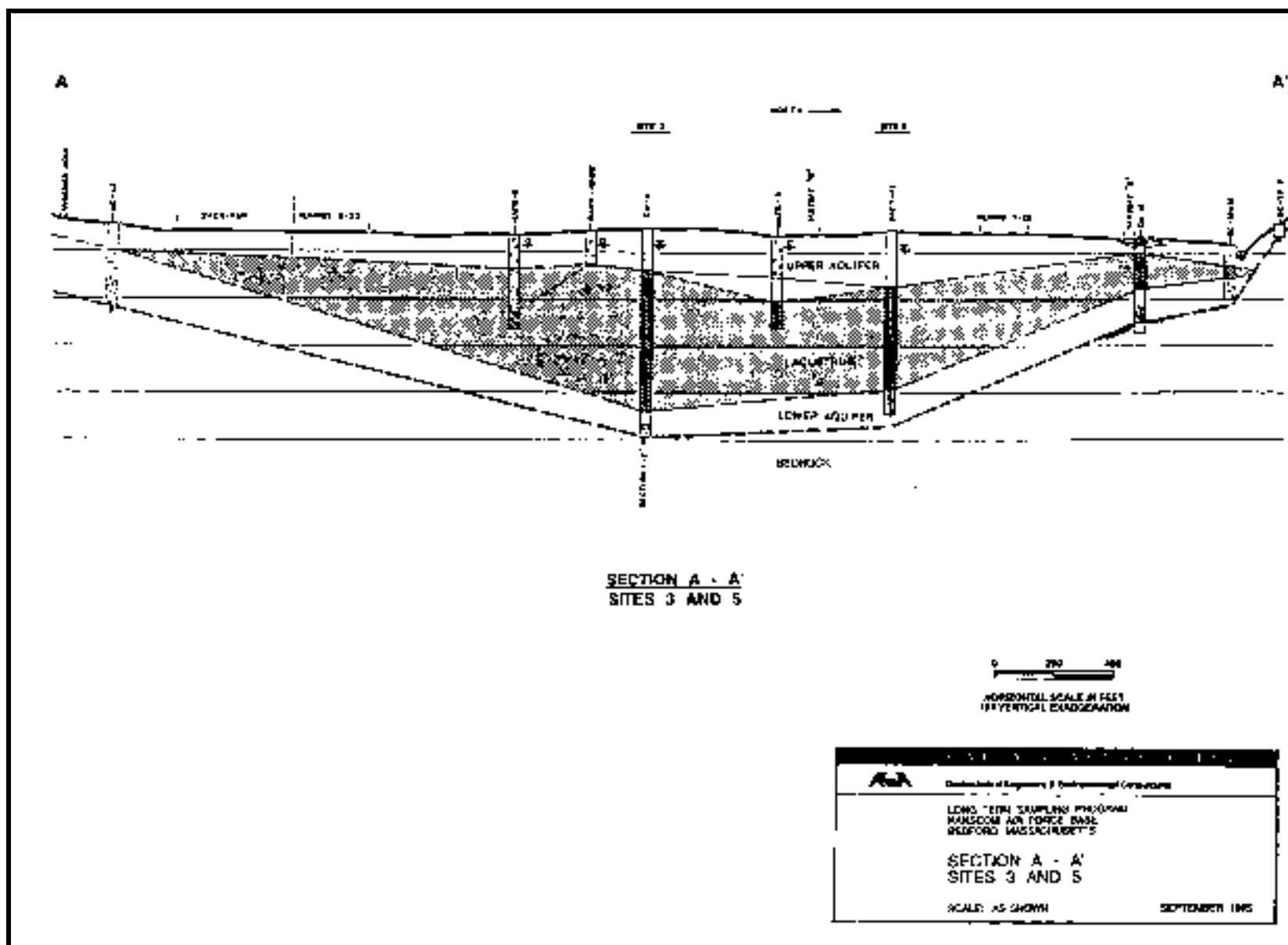


Figure 6. HAFB Site 3

SITE 1

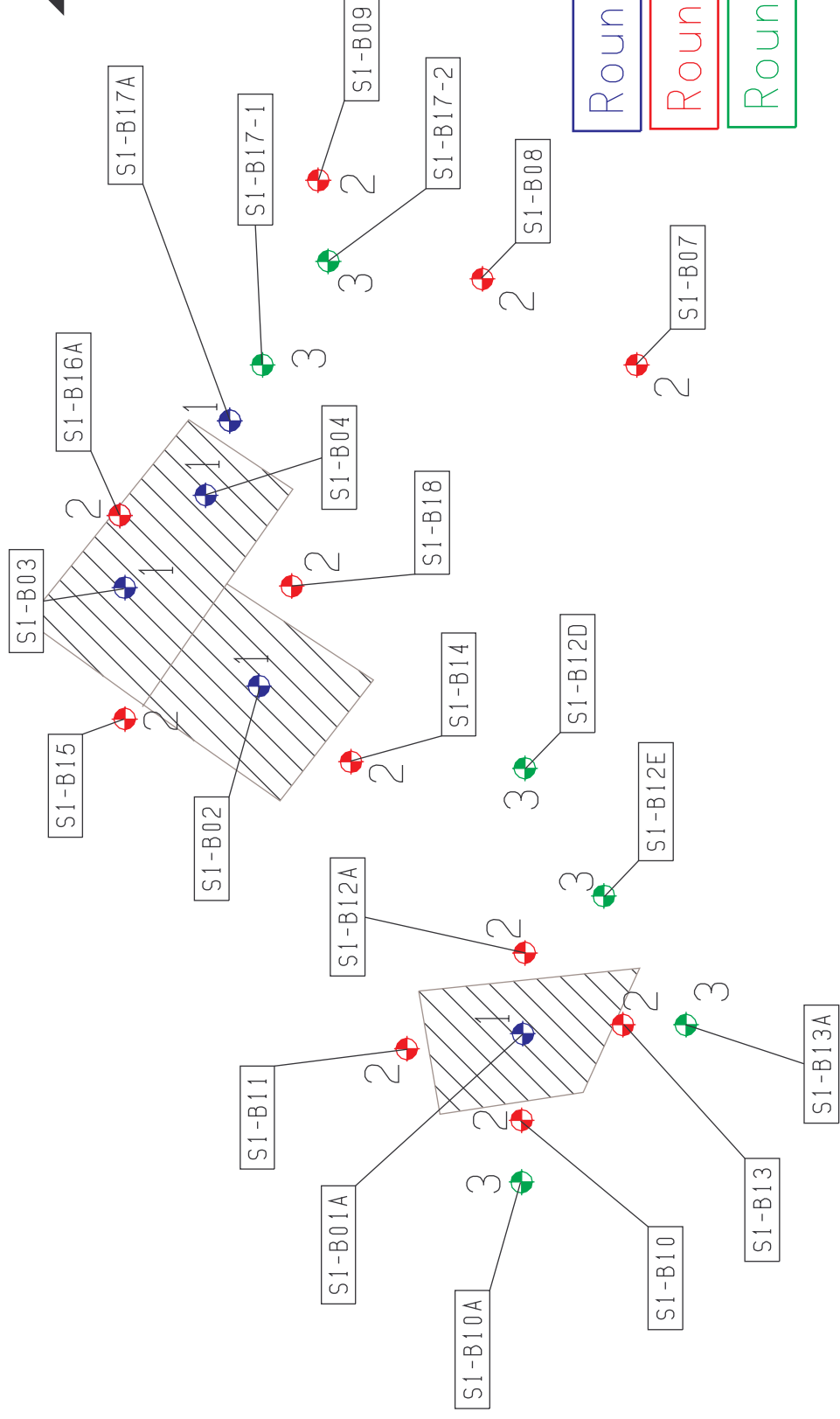
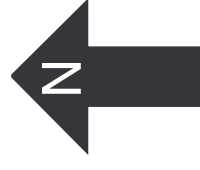
VOCs. Figure 7a shows the location of samples collected during Rounds 1, 2, and 3. Figures 7b-7e depict the chlorinated VOCs, benzene, toluene, ethylbenzene and xylenes (BTEX) contamination at depths above and below the 4-ft fire training burn pit bottoms. Contaminant concentrations are shown with respect to their 10DAF concentrations as measured by quantitative GC/MS. For example, cis-1,2-dichloroethene is compound #5 in the Figure 4 table. Figure 7d shows that cis-1,2-dichloroethene was detected in soil from boring S1-B17A at depths between 5.75 and 8-ft. It is listed as S1-B17A-(5.75-08): #5 4-10DAF. The 4-10DAF shows that this target compound was present at four times the 10DAF concentration or 800-ppb. This notation was used to facilitate the decisions made in the field and to quickly visualize the contamination at the quantitation limit. Recall that the action level for Operable Unit 1 was set at the 20DAF soil screening level for evaluating contaminant risk to ground water.

Whenever a triangle is shown in the map, it indicates non detectable (ND) target compound concentrations by rapid screening GC/MS at the specified interval. In every instance for Site 1 another soil sample from the same boring at another depth was analyzed by quantitative GC/MS and found to contain no detectable VOCs. As an example, samples from S1-B05, S1-B07, S1-B08, S1-B17-2, and S1-B09 were found to contain no detectable VOCs above the burn pits by screening GC/MS, see Figures 7b and 7c. Subsequent confirmation was made by quantitative GC/MS below the pit bottom, see Figures 7d and 7e. A square indicates target compound concentrations less than 10DAF or at non-detectable levels as determined by quantitative GC/MS. In contrast, circles show samples where quantitative GC/MS measured target compound concentrations were greater than the corresponding 10DAF level.

Although samples were collected from 23 boring locations, only five samples from three borings (S1-B04, S1-B10A, and S1-B17A) contained detectable target compounds above their 10DAF concentration. A total of 210 soil samples was screened from which 51 samples were analyzed by quantitative GC/MS. All areas positively identified to be contaminated were encircled by soil samples from borings shown to contain non detectable target compound concentration except S1-B10A-(4.5-6) which contained cis-1,2-dichloroethene and trichloroethene at levels between 10DAF and 20DAF. Therefore, the core technical team decided that no additional Round 3 samples required analysis to bound this location.

Semi-VOCs No maps were produced for PCBs and PAHs. A total of 46 samples was collected and analyzed by quantitative TDGC/MS. Only one sample, S1-B04-(6.2-8), contained detectable levels of PAH: acenaphthene 1-ppm, acenaphthylene 0.7-ppm, fluorene 2-ppm, anthracene/phenanthrene 5-ppm, pyrene 1-ppm, fluoranthene 0.8-ppm, and benzo(a)anthracene/chrysene 2-ppm. No samples contained detectable levels of PCBs. Since the reporting limits for PAH were well below the 20DAF concentrations and the fact that only one burn pit sample contained any appreciable PAH, no additional samples were collected or analyzed.

Site 1



Scale



2.5x vertical exaggeration

Figure 7a.

HAFB: Site 1 - Chlorinated VOCs above 4ft

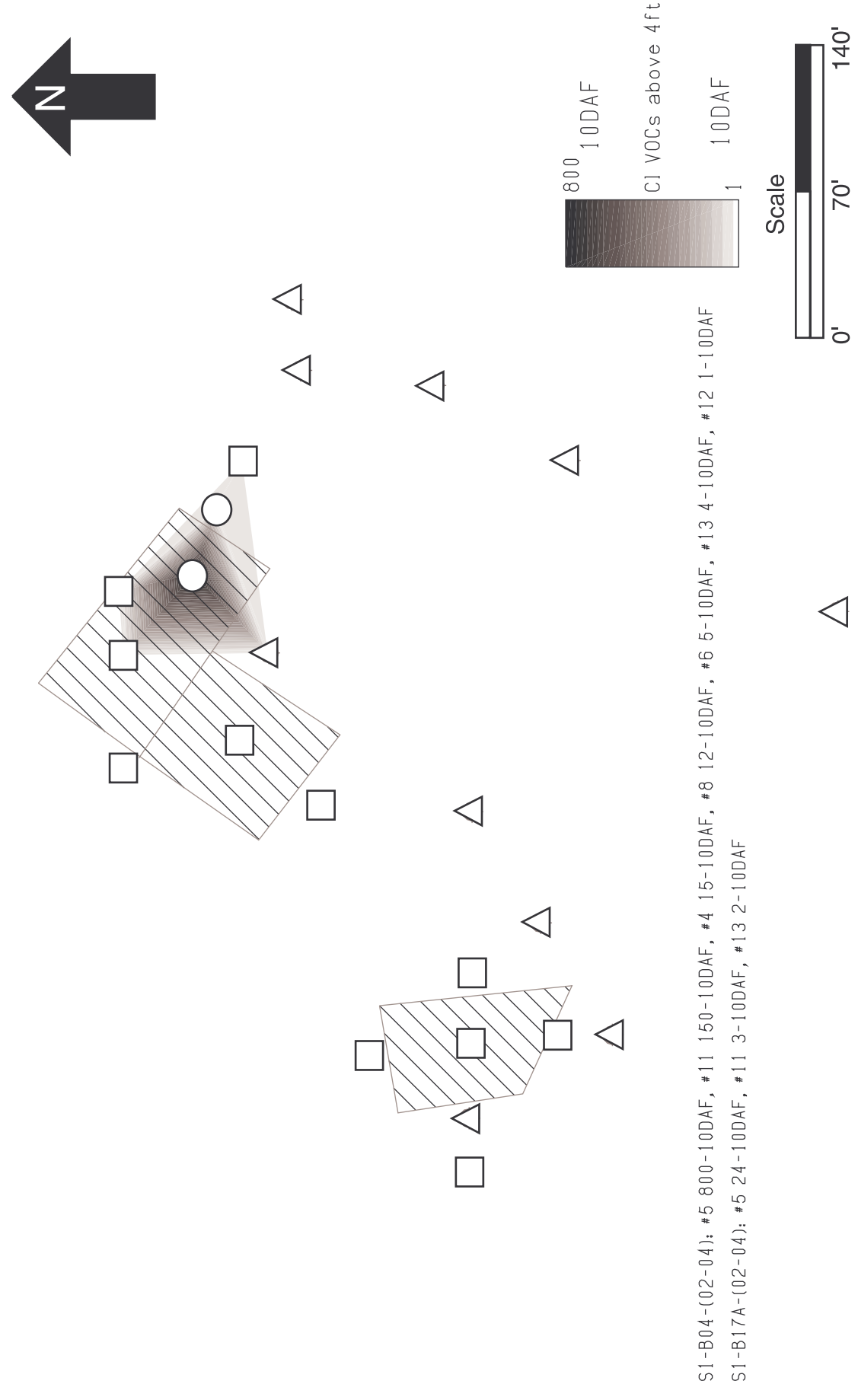
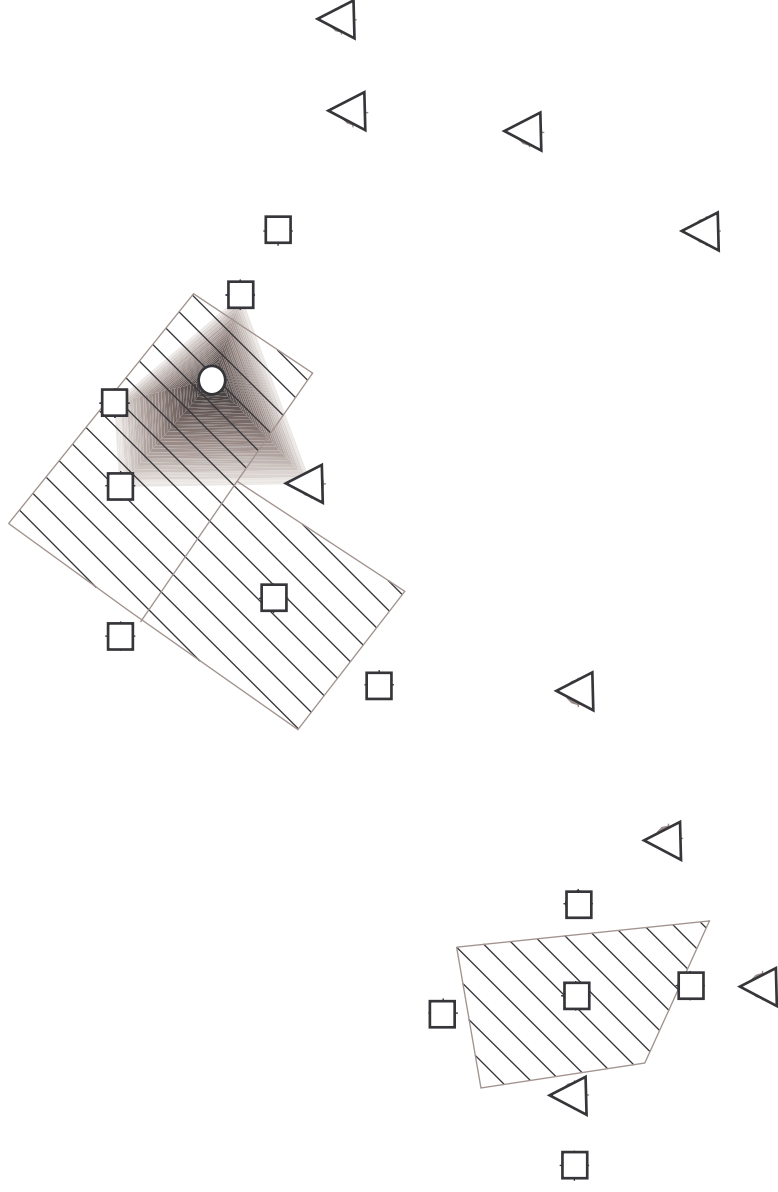
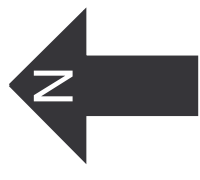


Figure 7b.

HAFB: Site 1 - BTEX above 4ft



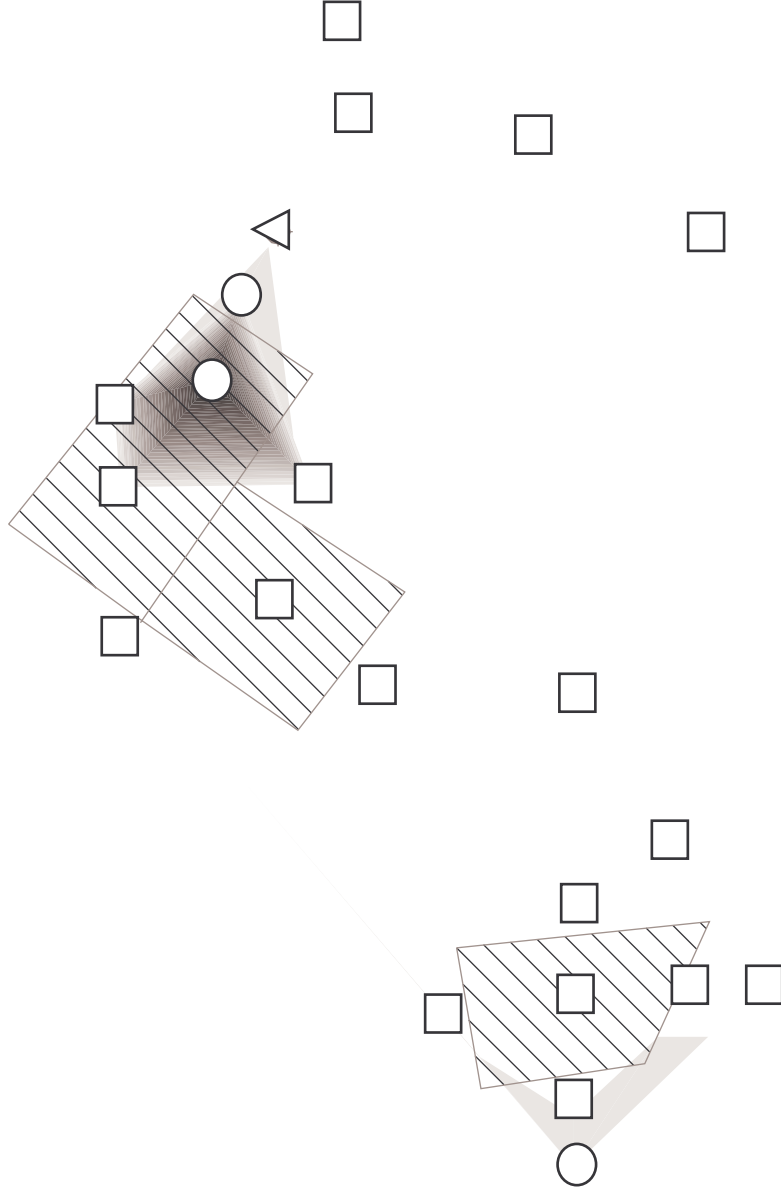
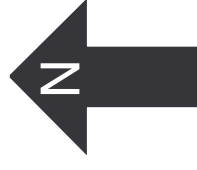
S1-B04-(02-04): #5 800-10DAF, #11 150-10DAF, #4 15-10DAF, #8 12-10DAF, #6 5-10DAF, #12 1-10DAF
 S1-B17A-(02-04): #5 24-10DAF, #11 3-10DAF, #13 2-10DAF



2.5x vertical exaggeration

Figure 7c.

HAFB: Site 1 - Chlorinated VOCs below 4ft



S1-B04-(6.2-8): #11 330-10DAF, #5 200-10DAF, #4 13-10DAF, #13 11-10DAF, #8 10-10DAF, #6 5-10DAF, #12 1.1-10DAF
 S1-B10A-(4.5-06): #11 2-10DAF, #5 1.5-10DAF
 S1-B17A-(5.75-08): #5 4-10DAF

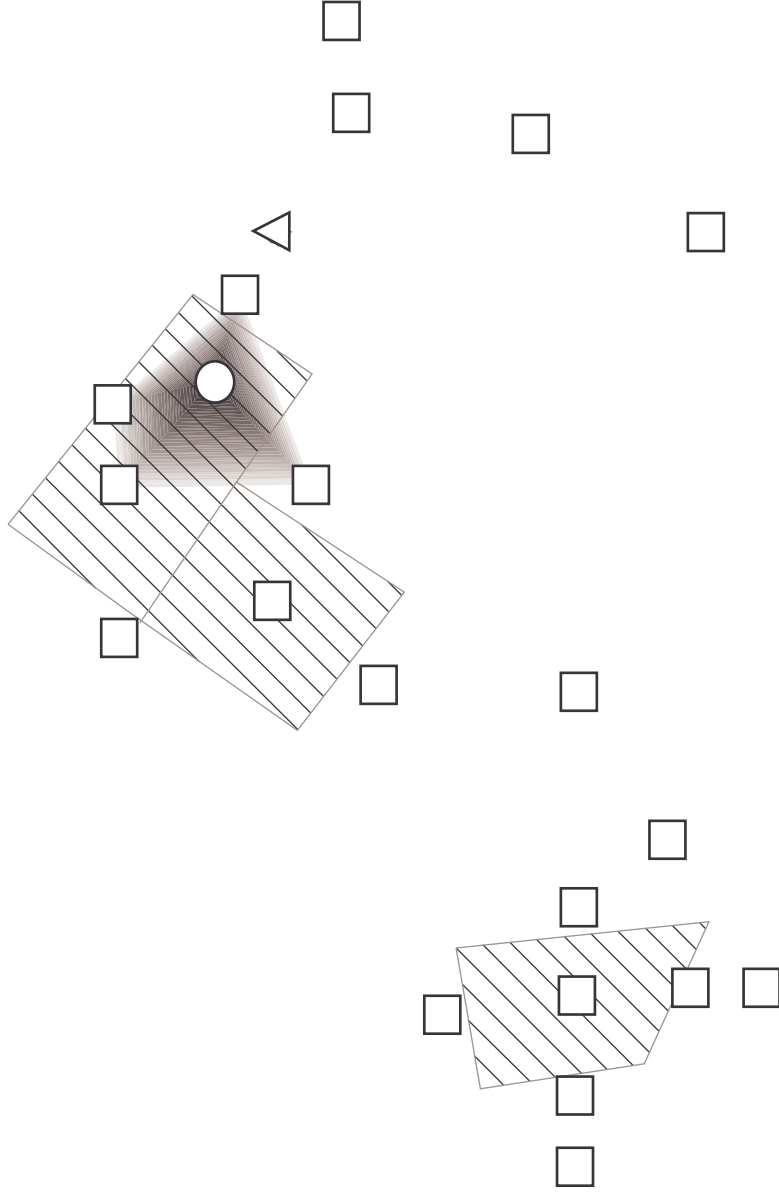
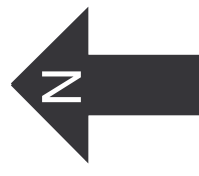
Scale



2.5x vertical exaggeration

Figure 7d.

HAFB: Site 1 - BTEX below 4ft



S1-B04-(6.2-8): #11 330-10DAF, #5 200-10DAF, #4 13-10DAF, #13 11-10DAF, #8 10-10DAF, #6 5-10DAF, #12 1.1-10DAF
 S1-B10A-(4.5-06): #11 2-10DAF, #5 1.5-10DAF
 S1-B17A-(5.75-08): #5 4-10DAF

Scale



2.5x vertical exaggeration

Figure 7e.

Metals No maps were produced for metals. Soil samples were analyzed above and below the pit bottoms. Field ICP/OES analysis yielded no detectable lead or cadmium concentrations above their respective 10DAF concentrations; namely, 200-ppm and 4-ppm respectively. The total number of soil samples analyzed was twenty-two, fourteen samples had concentrations between 10 and 40-ppm lead while twelve samples produced concentrations between 0.35-ppm and 0.71-ppm cadmium. After analyzing seven of the 38 samples collected in Round 2, EPA, Air Force and Tufts staff agreed that none of the remaining samples required analysis.

SITE 2

Situated on this site is a recharge basin whose elevation is 6-ft above ground level. Sample IDs within the recharge basin include the 6-ft elevation depth, while those samples outside the recharge basin depict depth from ground level.

VOCs Soil samples were collected from 18 borings to subsurface depths of 16-ft, Figure 8a. A total of 177 sample locations was screened from these borings. Samples were collected for quantitative GC/MS analysis from one-half of these locations yielding 58 soil samples. Of the eighteen borings seven borings (S2-B01, S2-B02, S2-B03, S2-B04, S2-B13, S2-B15, S2-B15A) were contaminated at twelve different sample locations. Target compound concentrations were greater than 10DAF. Tetrachloroethene is present in the soil between 42 and 540-ppb in three borings (S2-B01, S2-B02, S2-B04) above 8-ft (i.e., depth from ground level), see Figure 8b. In contrast, eight chlorinated solvents were found below 8-ft, see Figure 8d. The three solvents with the highest concentrations were tetrachloroethene at percent levels (estimated, S2-B15), 1,1,1-trichloroethane at 15-ppm (S2-B02) and cis-1,2-dichloroethene at 15-ppm (S2-B13). Figures 8c and 8e reveal toluene as the only BTEX contaminant at levels above 10DAF in the pits: 24 to 42-ppm between the depths of 7-ft and 12-ft. Benzene was found in only one boring (S2-B15) at 8 to 10-ft. No detectable amounts of benzene were found in borings S2-B15A or S2-B04, two very closely sampled locations. All pit locations shown to contain target compounds in Round 1 greater than 10DAF were encircled by the collection of soil samples in Rounds 2 and 3. Two samples were collected away from the recharge basin to assess if contaminant migration through soil may be occurring. No detectable levels of VOCs were found by either screening or quantitative GC/MS down to subsurface depths of 16-ft.

Semi-VOCs No maps were made for PCBs and PAHs. Historical information at this site indicated that PCB/PAH analysis was not necessary. During the investigation, it became evident that some soil locations were contaminated by a highly viscous petroleum product. Soils were initially selected for analysis based on visible contamination. Positive identification was made in three Round 1 samples: drum pit S2-B04 at two depths (13 to 15-ft and 19 to 21-ft) and S2-B15 (14 to 16-ft). Positive identification was made in one second round sample S2-B13 at a depth of 14 to 16-ft. The highest PAH concentrations found in all soil samples analyzed and their action levels (20DAF) were benzo(a)anthracene 0.3-ppm (2-ppm), benzo(b)fluoranthene 0.5-ppm (5-ppm), benzo(a)pyrene 1-ppm (8-ppm), and dibenzo(a,h)anthracene 0.3-ppm (2-ppm). All PAH detected were well below their 20DAF concentrations. Based on results from 12 of the 24 samples collected, a decision was made not to analyze the remaining samples.

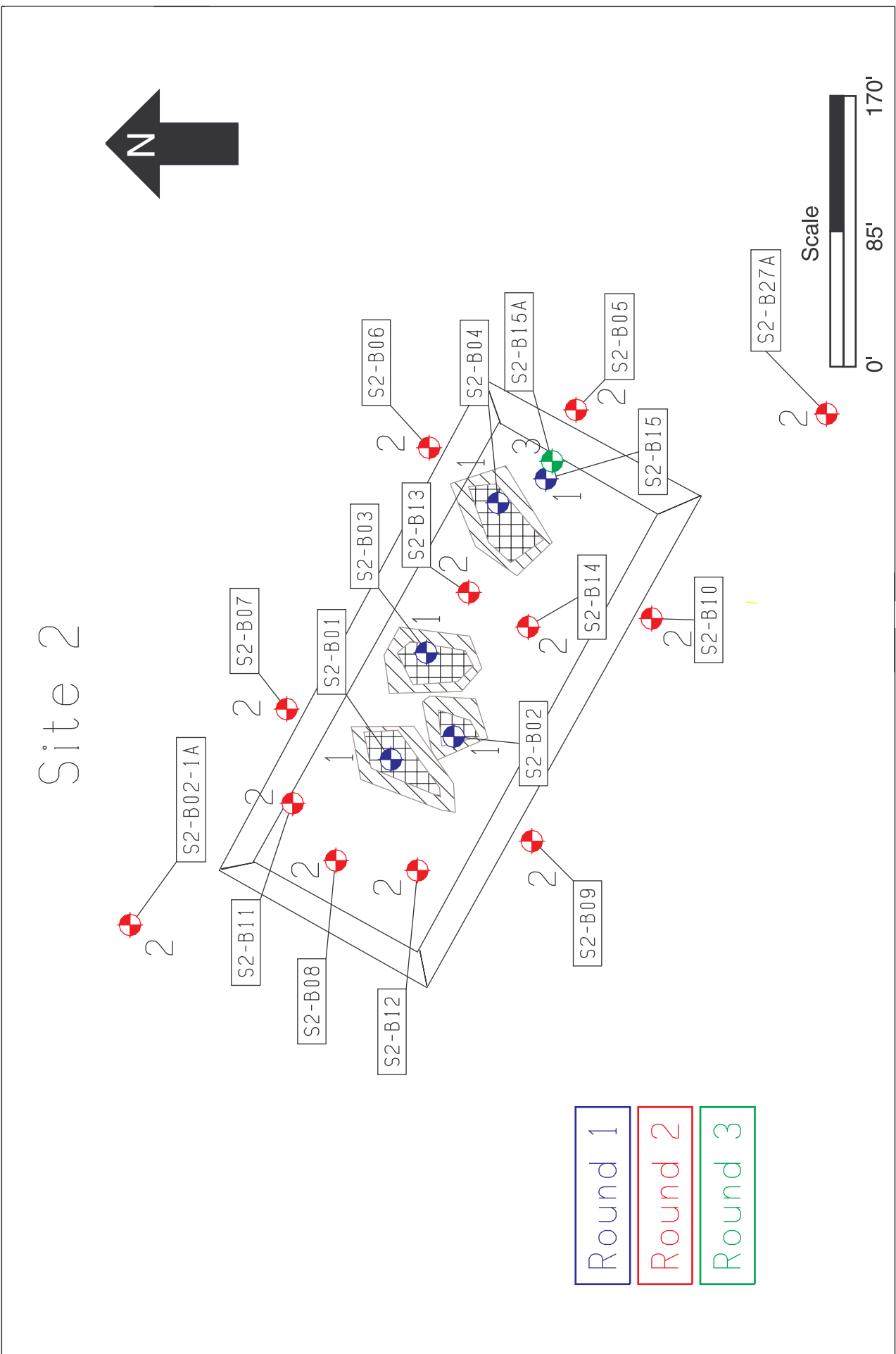
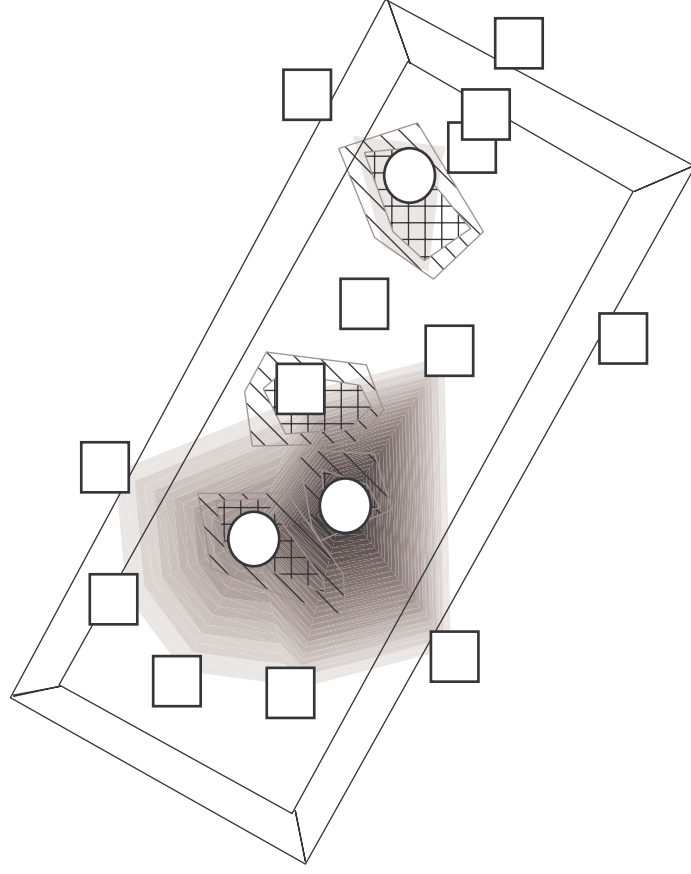


Figure 8a.

HAFB: Site 2 - Chlorinated VOCs above 8ft



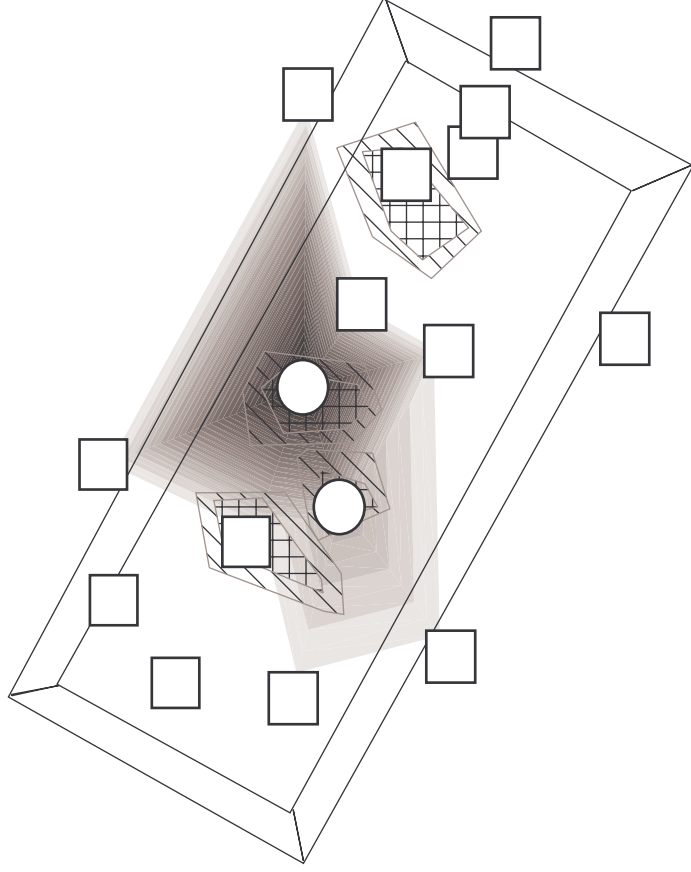
- S2-B01-(12-14): #13 5-10DAF
- S2-B02-(13-15): #13 18-10DAF, #14 1.5-10DAF
- S2-B03-(13-15): #14 4-10DAF
- S2-B04-(13-15): #13 1.4-10DAF



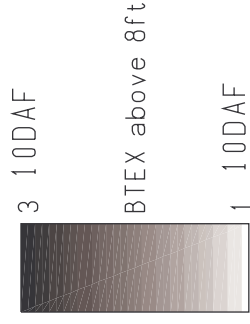
Scale

0' 85' 170'

Figure 8b.



S2-B01-(12-14); #13 5-10DAF
 S2-B02-(13-15); #13 18-10DAF, #14 1.5-10DAF
 S2-B03-(13-15); #14 4-10DAF
 S2-B04-(13-15); #13 1.4-10DAF

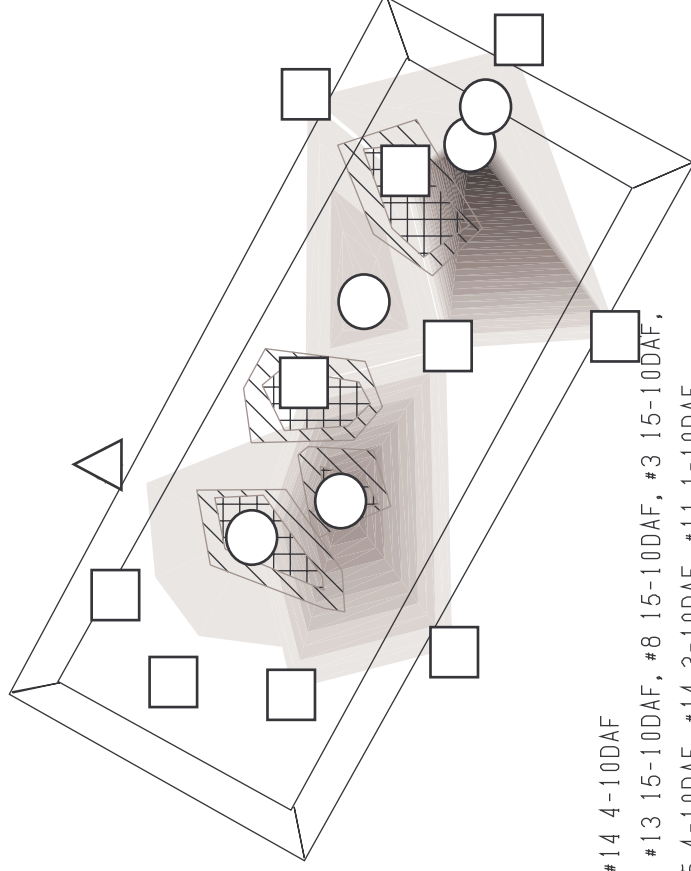


Scale

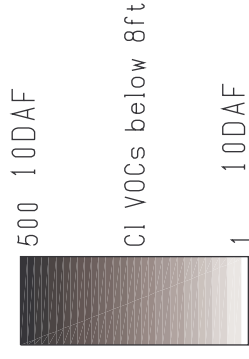


Figure 8c.

HAFB: Site 2 - Chlorinated VOCs below 8ft



- S2-B01-(20-22): #13 4-10DAF, #14 4-10DAF
 S2-B02-(16-18): #4 160-10DAF, #13 15-10DAF, #8 15-10DAF, #3 15-10DAF,
 #9 7-10DAF, #5 4-10DAF, #14 3-10DAF, #11 1-10DAF
 S2-B02-(20-22): #14 7-10DAF, #4 4-10DAF, #13 4-10DAF, #5 3-10DAF, #3 1-10DAF
 S2-B03-(16-18): #14 1.6-10DAF
 S2-B13-(14-16): #5 1.9-10DAF, #13 1.4-10DAF
 S2-B13-(18-20): #5 75-10DAF, #3 1.7-10DAF, #4 1.5-10DAF
 S2-B15-(14-16): #11 27000-10DAF, #12 9-10DAF, #13 9-10DAF, #5 2-10DAF, #6 1.5-10DAF
 S2-B15A-(18-20): #5 33-10DAF, #3 1.1-10DAF

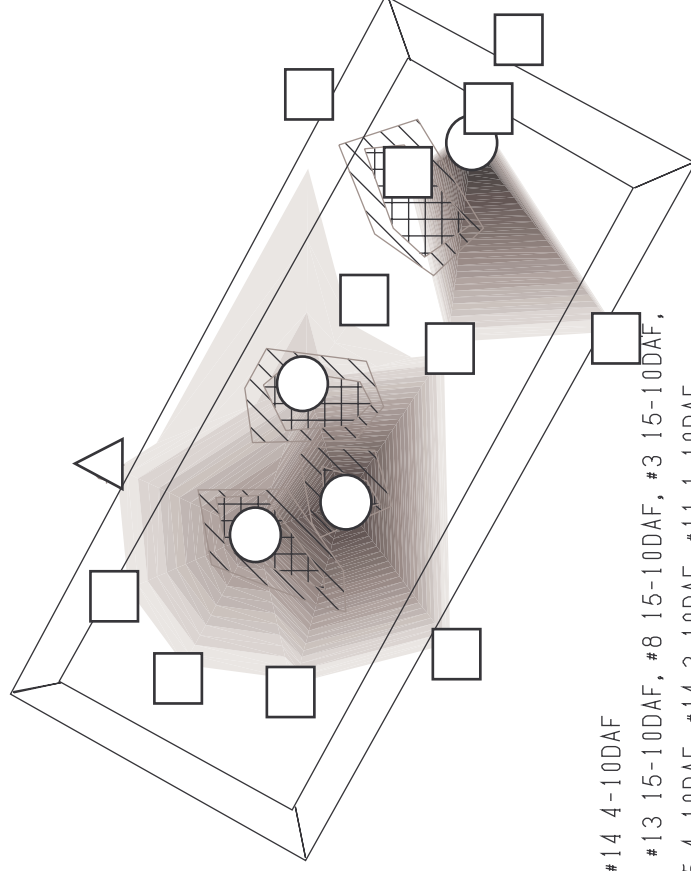


Scale

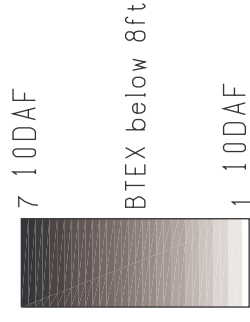


Figure 8d.

HAFB: Site 2 - BTEX below 8ft



- S2-B01-(20-22): #13 4-10DAF, #14 4-10DAF
 S2-B02-(16-18): #4 160-10DAF, #13 15-10DAF, #8 15-10DAF, #3 15-10DAF,
 #9 7-10DAF, #5 4-10DAF, #14 3-10DAF, #11 1-10DAF
 S2-B02-(20-22): #14 7-10DAF, #4 4-10DAF, #13 4-10DAF, #5 3-10DAF, #3 1-10DAF
 S2-B03-(16-18): #14 1.6-10DAF
 S2-B13-(14-16): #5 1.9-10DAF, #13 1.4-10DAF
 S2-B13-(18-20): #5 75-10DAF, #3 1.7-10DAF, #4 1.5-10DAF
 S2-B15-(14-16): #11 27000-10DAF, #12 9-10DAF, #13 9-10DAF, #5 2-10DAF, #6 1.5-10DAF
 S2-B15A-(18-20): #5 33-10DAF, #3 1.1-10DAF



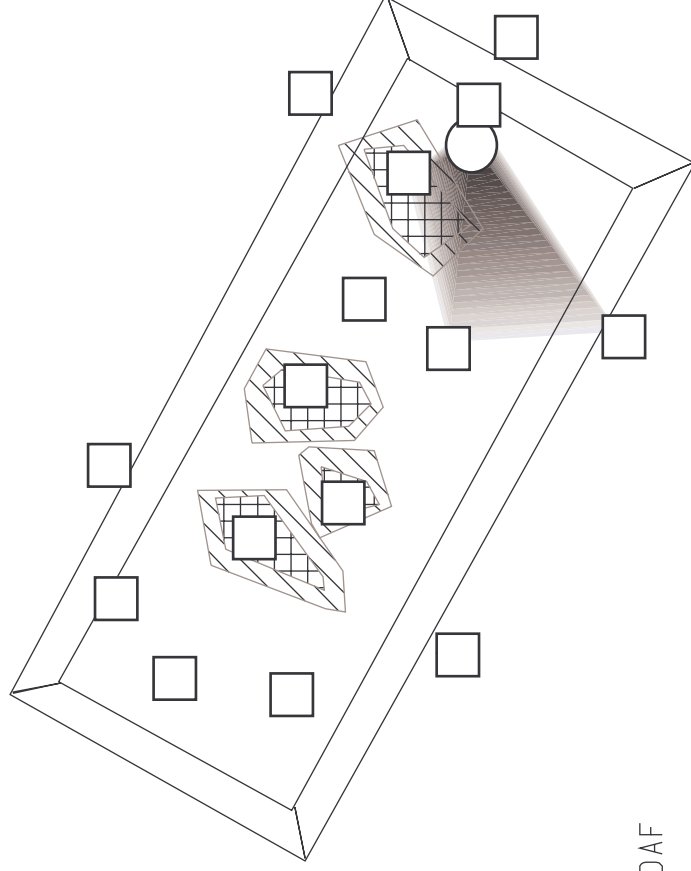
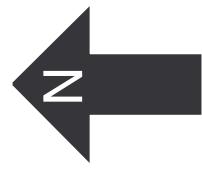
Scale



0' 85' 170'

Figure 8e.

HAFB: Site 2 - Cadmium



S2-B01-(12-14): Pb 1.3-10DAF

S2-B04-(13-15): Pb 1.1-10DAF

S2-B15-(14-16): Pb 1.8-10DAF, Cd 1.6-10DAF

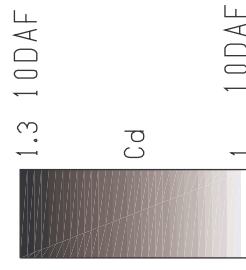
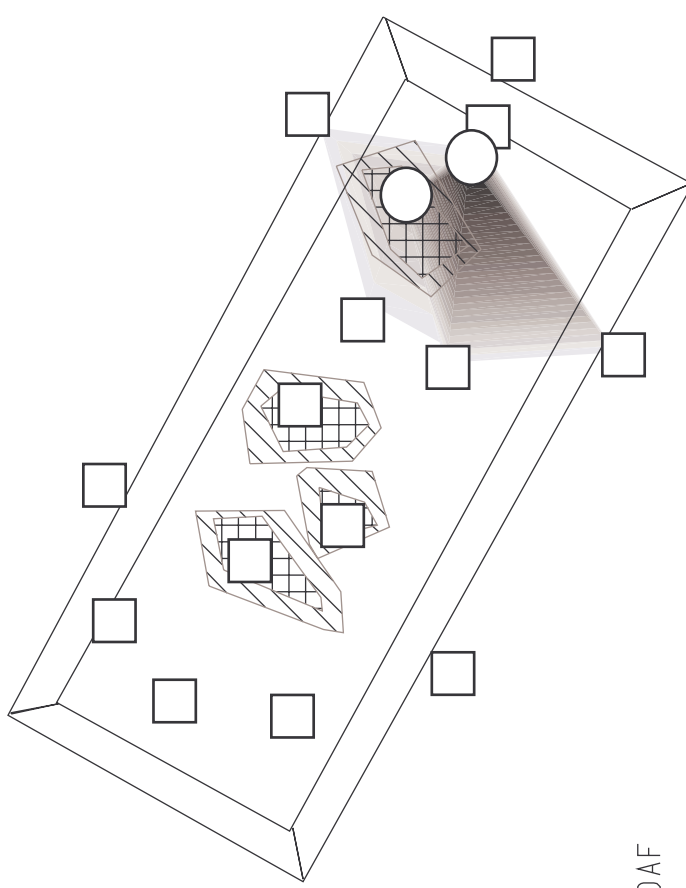
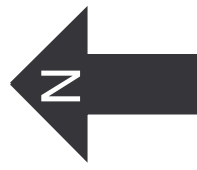


Figure 8f.

HAFB: Site 2 - Lead



S2-B01-(12-14): Pb 1.3-10DAF
S2-B04-(13-15): Pb 1.1-10DAF
S2-B15-(14-16): Pb 1.8-10DAF, Cd 1.6-10DAF

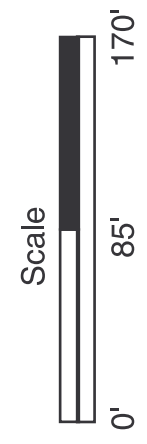
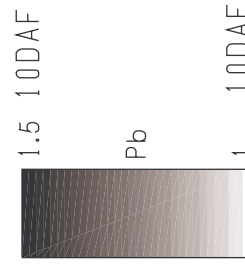


Figure 8g.

Metals Figures 8f and 8g depict the cadmium and lead concentrations for Site 2. The pit samples (S2-B01, S2-B02, S2-B03, S2-B04) had the highest concentrations of lead and cadmium. For lead the concentrations ranged between 10-ppm and 370-ppm at or near the pit bottoms while cadmium concentrations were between 0.35-ppm and 1-ppm. Outside the pits the concentrations dropped dramatically to levels between ND and 50-ppm for lead and non detectable levels for cadmium. Boring sample S2-B15 (14-16) was the exception with 360-ppm lead and 6-ppm cadmium. All samples were below the site-specific 20DAF levels of concern. After reviewing data from 54 of the 68 samples collected, it was decided that no additional sample analysis was needed.

SITE 3

Situated on this site is a recharge basin whose elevation is 6-ft above ground level. Sample IDs within the recharge basin include the 6-ft elevation depth, while those samples outside the recharge basin depict depth from ground level.

VOCs Figure 9a shows the boring locations sampled during Rounds 1, 2, and 3. Soil from 25 boring locations was analyzed to subsurface depths of approximately 16-ft. Screening analyses were performed on 214 samples from these borings with 49 analyses performed by quantitative GC/MS. Figures 9b-9c illustrate the results for samples collected and analyzed above 8-ft (i.e., depth from ground level). All of the BTEX compounds were found in soil samples collected from boring S3-B01; 1.3-ppm, 3,100-ppm, 71-ppm, and 210-ppm, respectively. At the same location cis-1,2-dichloroethene was also detected at 2.8-ppm. Contamination was found in samples collected from S3-B06 (tetrachloroethene at 60-ppb) and S3-B08 (cis-1,2-dichloroethene at 800-ppb). Analysis of Round 2 samples from all elevations surrounding the two borings revealed no detectable VOCs. No chlorinated solvent contamination was found above the 20DAF level at depths below 8-ft. Toluene was the only BTEX constituent detected (18-ppm in S3-B05) below 8-ft and above the 20DAF (12-ppm) concentration. The analysis of soil samples surrounding all four pits where contamination was found yielded non detectable signals.

Semi-VOCs Based on historical information, no samples for this site were preselected for PAH and PCB analysis in the dynamic workplan. Soil samples were analyzed from borings where BTEX constituents were found and from pits where drums were buried. Ten samples were selected to verify the absence of PAHs and PCBs. Of the eight drum burial pits only two pits contained measurable contaminants. Total PCB concentrations in S3-B01-(13-15) and S3-B06-(10-12) were 3-ppm and 2-ppm, respectively. PAHs were also detected in S3-B06 below the 20DAF levels. No PAHs or PCBs were found in any other sample. Although total PCBs were found in two samples above the action level, the core technical team determined that no additional analyses were required to decide future vadose soil actions.

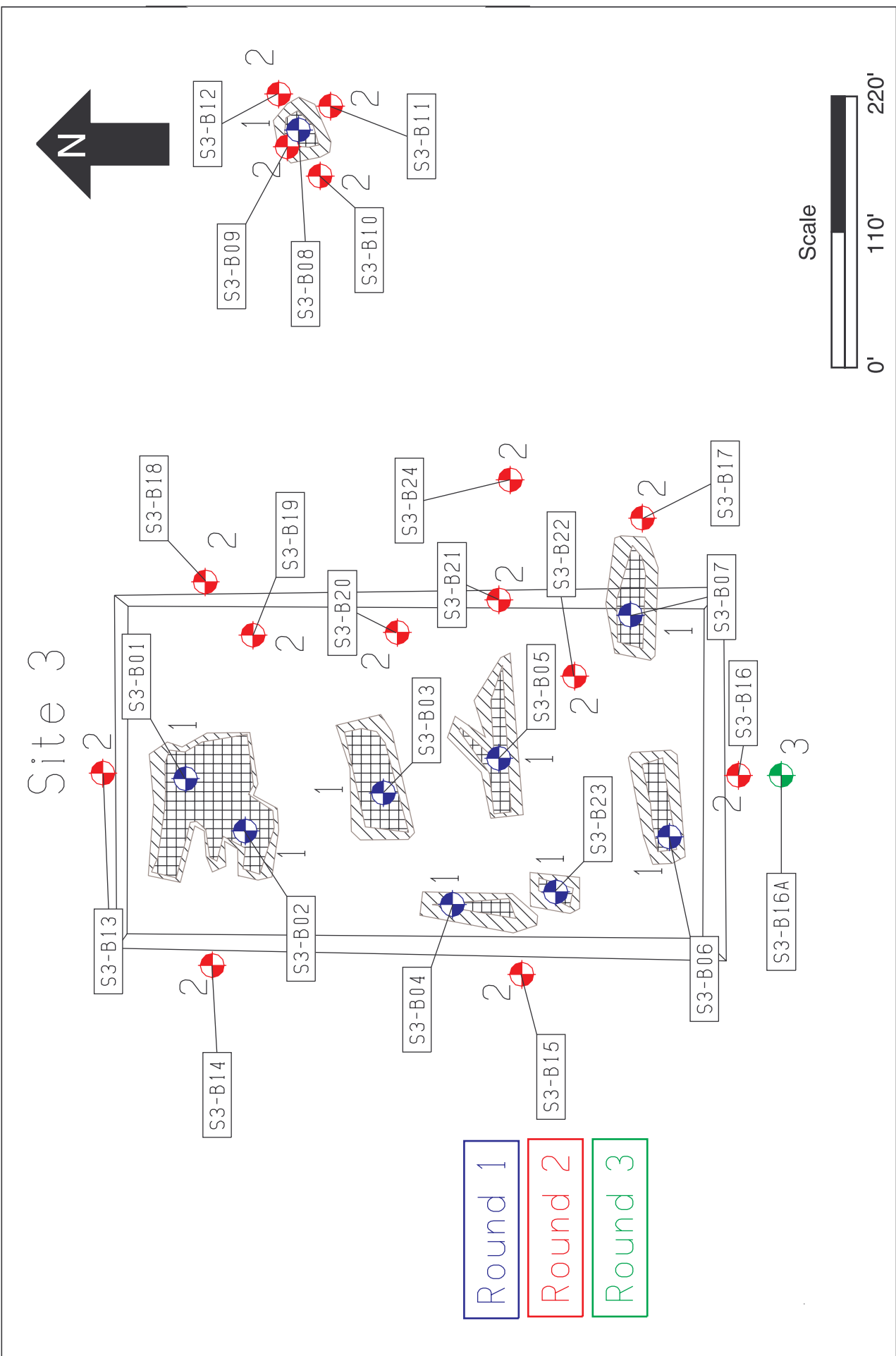


Figure 9a.

HAFB: Site 3 - Chlorinated VOCs above 8ft

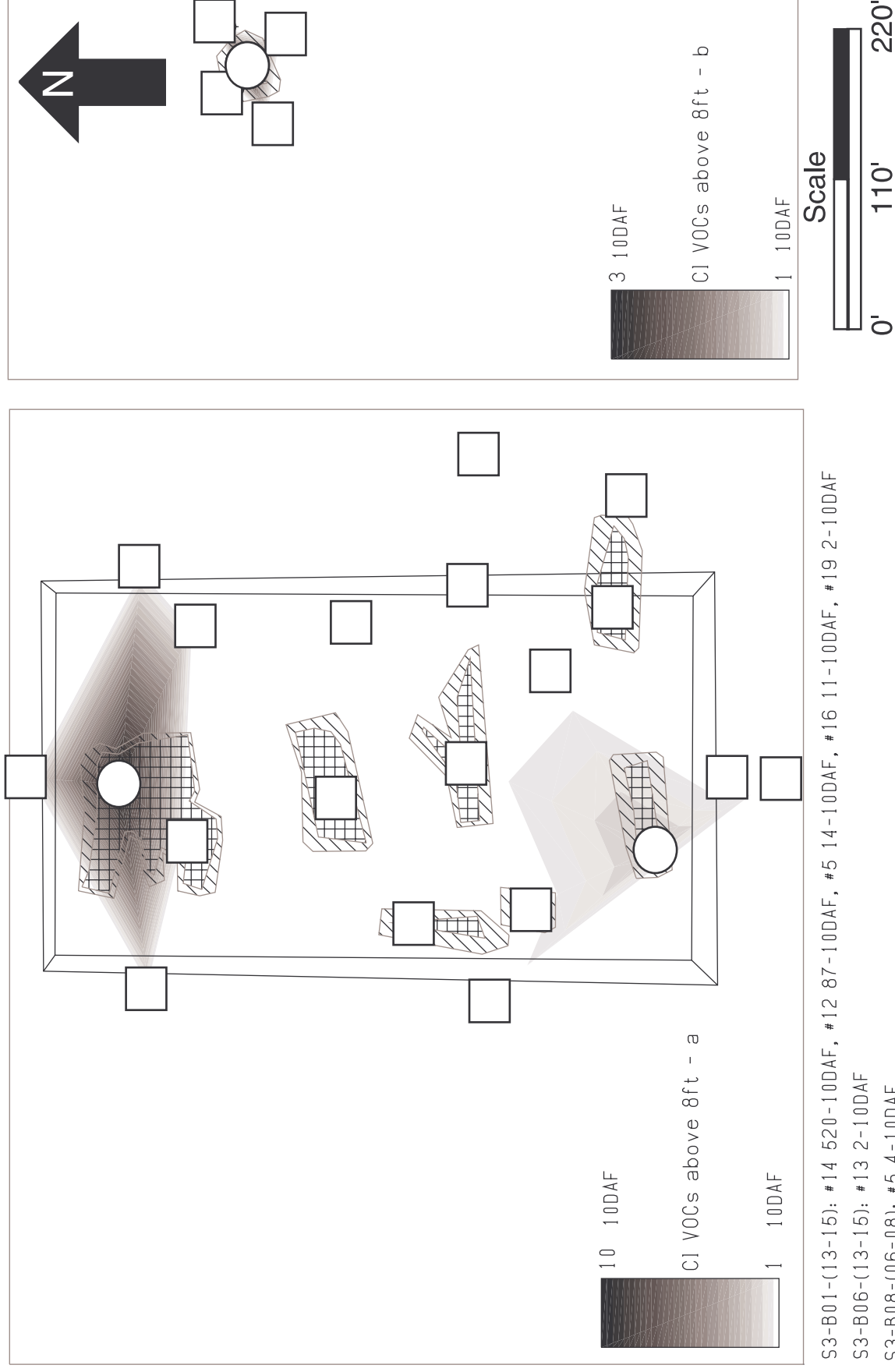
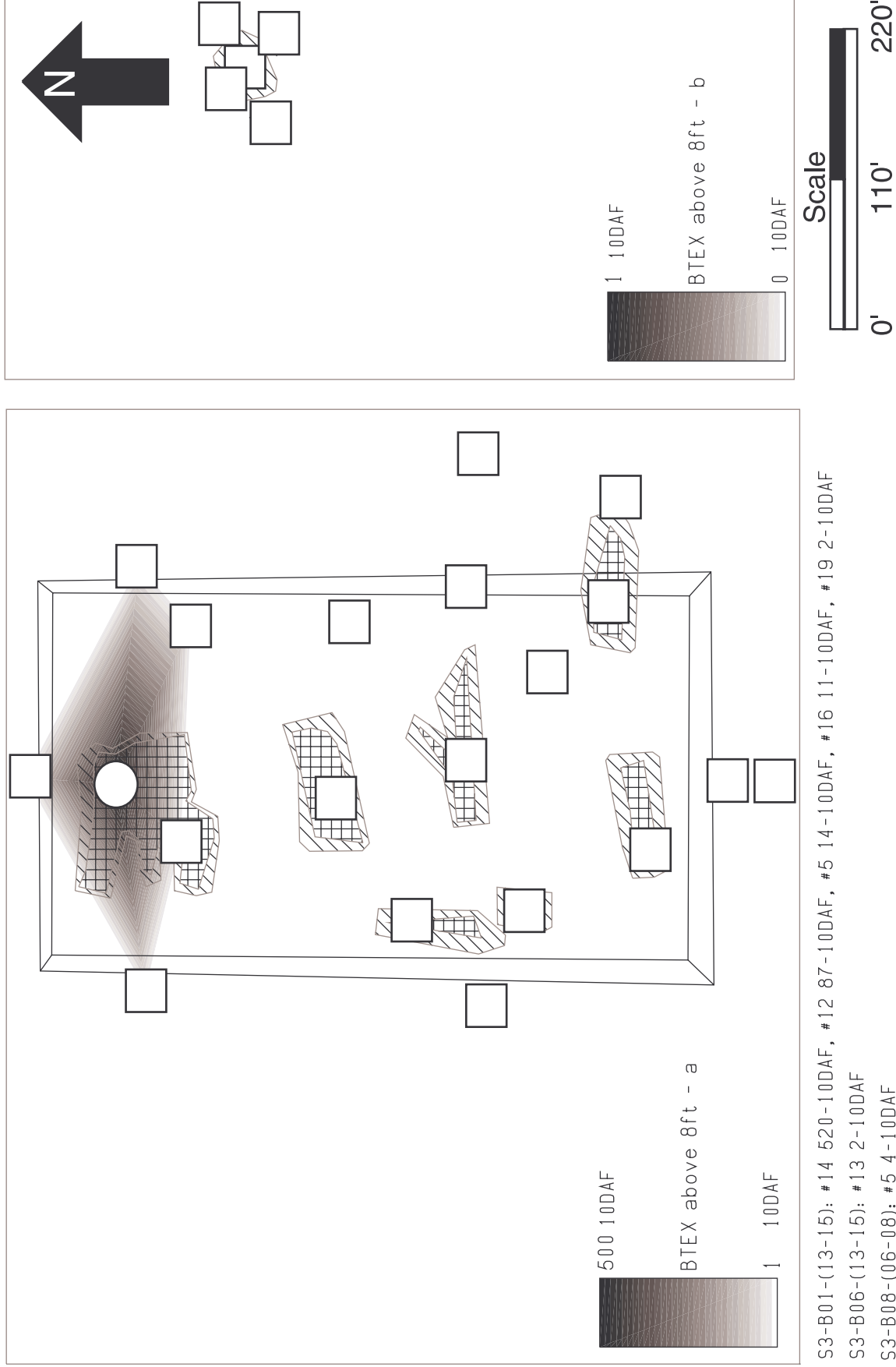


Figure 9b.



S3-B01-(13-15): #14 520-10DAF, #12 87-10DAF, #5 14-10DAF, #16 11-10DAF, #19 2-10DAF
S3-B06-(13-15): #13 2-10DAF
S3-B08-(06-08): #5 4-10DAF

Figure 9c.

HAFB: Site 3 - Chlorinated VOCs below 8ft

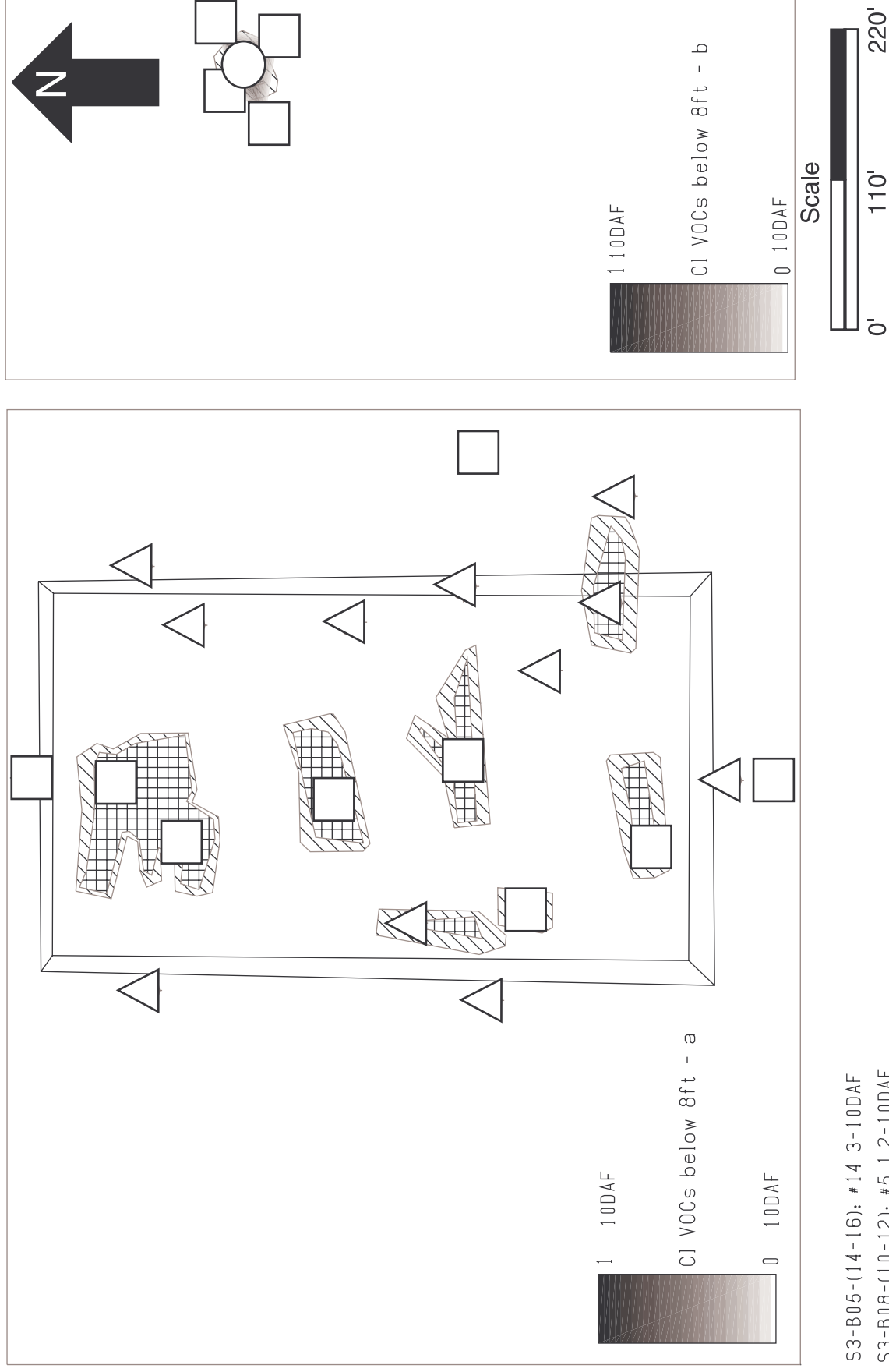
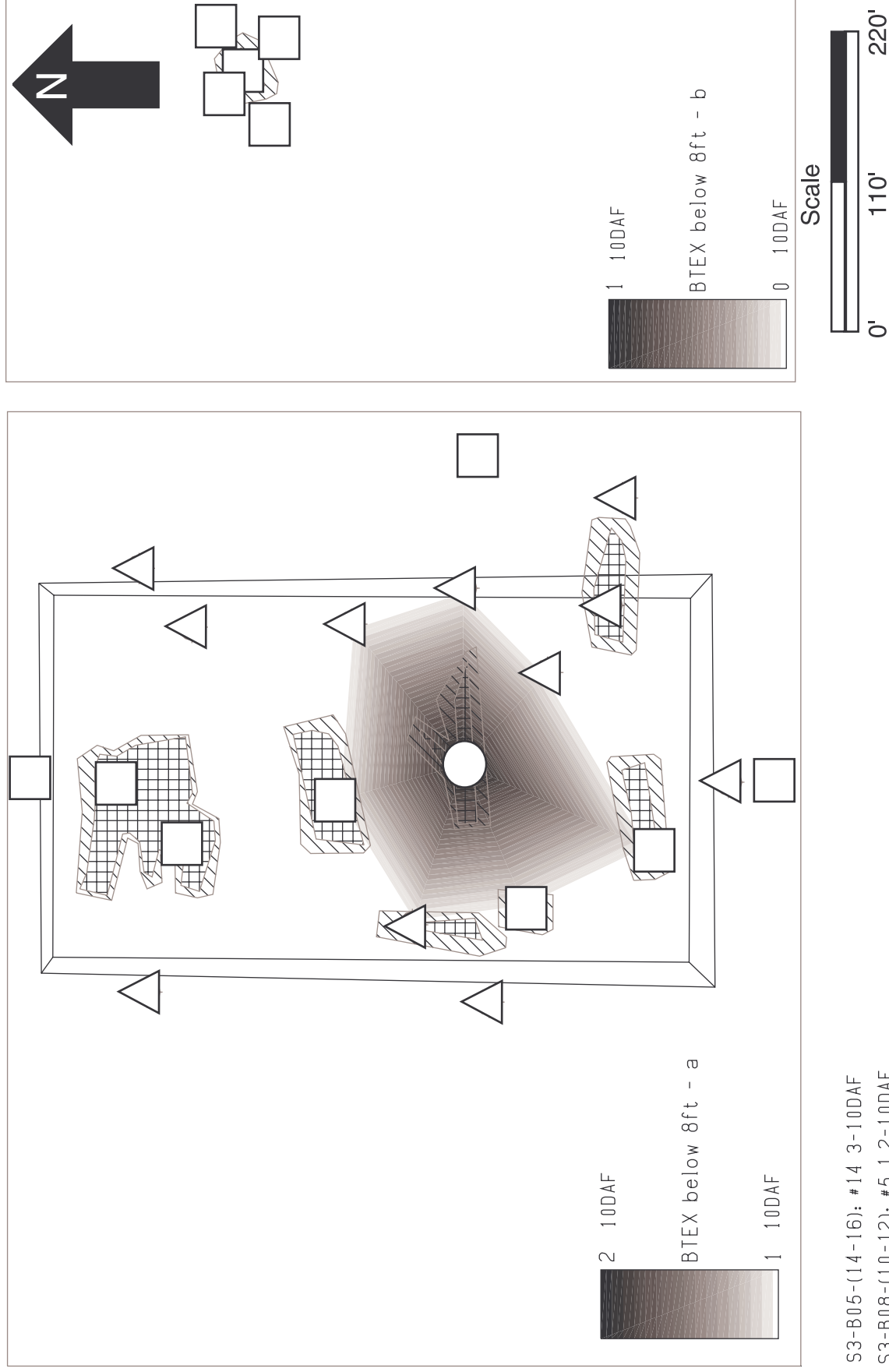


Figure 9d.

HAFB: Site 3 - BTEX below 8ft



S3-B05-(14-16): #14 3-10DAF
S3-B08-(10-12): #5 1.2-10DAF

Figure 9e.

HAFB: Site 3 - Cadmium

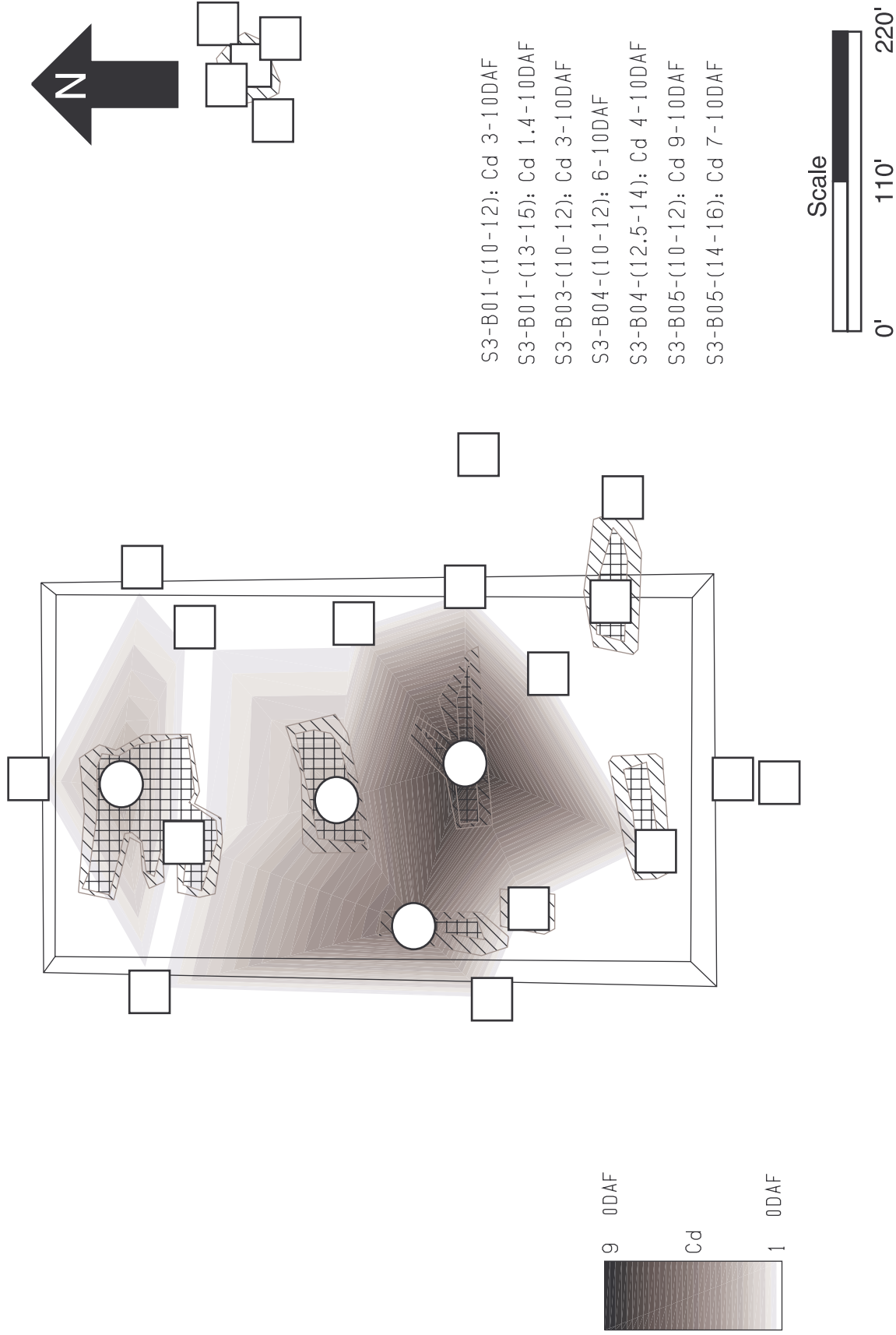


Figure 9f.

Metals Initially 53 samples were collected for this site. ICP/OES analysis was performed on 45 of these samples. Pit bottom concentrations for lead were less than 65-ppm and much less outside the pits. Cadmium concentrations at pit borings S3-B01, S3-B03, S3-B04, and S3-B05 were between 11-ppm and 36-ppm. Multiple samples at different depths above and below the pit bottoms were analyzed from each boring. Only at the pit bottoms were concentrations greater than 20DAF. To learn whether cadmium migration had occurred outside the pits at least one sample per boring was analyzed at the same depth cadmium was found inside the pit. The results of these analyses were ND, see Figure 9f. It appears that contamination is confined to the pit since non detectable levels were found outside the pit.

4.1.1 Summary of Findings

The adaptive sampling and analysis program provided information on a “real-time” basis to support on-site decision making. Both the screening and quantitative data met the data quality objectives of the project. Contaminated soil volumes were conservatively estimated for VOCs at each site. Quantitative data was used to determine which borings contained contamination above or below the action level. For each boring, the screening data were used to estimate the vertical distance between points of contamination and non measurable levels. (Recall that screening analyses were made at 1-ft intervals, where possible.) Thus, the x-z and x-y coordinates were determined by using a combination of quantitative and screening data. From this, contaminated soil volumes were estimated by linearly interpolating between soil concentrations above the action level and non measurable levels for each x-z and x-y coordinate. Approximately 28,000-ft³, 243,000-ft³, and 66,000-ft³ of soil are estimated to be contaminated for Sites 1, 2, and 3; see Appendix IV.

Screening data showed the presence of target compounds in three of 23 borings in Site 1 with quantitative data verifying the same three borings. Moreover, both screening and quantitative data for borings S1-B4 and S1-B17A revealed the same contamination profiles. Namely, the presence of some chlorinated solvents at high concentrations with low levels of some BTEX constituents. No detectable VOCs were found between the surface and 4-ft by either analysis for boring S1-B10A where as both techniques identified low levels of trichloroethene at the 20DAF concentration below 4-ft. No additional quantitative analysis was done since the screening GC/MS response indicated no detectable organics at depths below 6-ft.

Contaminants above 10DAF were found in seven of the eighteen borings in Site 2. Overall, BTEX was found at or near the water table in medium to high concentrations, with chlorinated solvents more evenly distributed but also more concentrated at the lower depths. For instance, for boring S2-B02 both screening and quantitative data indicated increasingly higher levels of chlorinated solvents from 14-ft and down as well as fairly high levels of toluene at 19-ft and deeper. For boring S2-B15 screening data for many of the 4-ft tube sections was not possible. Soil concentrations overloaded the Bruker MS, requiring a 15 to 30-min bakeout period before analyzing the next sample. Screening data, given to the quantitative field laboratory staff, provided a good estimate for sample dilution. This reduced the number of samples requiring re-analysis by quantitative GC/MS and, at the same time, protected the MS. Quantitative GC/MS analysis indicated percentage levels of trichloroethene in the soil. Data for boring S2-B15A, only a few feet away, showed elevated levels of cis-1,2-dichloroethene at 18 to 20-ft and non detectable levels of trichloroethene at all depths by both screening and quantitative analysis.

Of the twenty-five borings in Site 3, only four of them contained target compounds greater than their 10DAF concentration. BTEX compounds were found in borings S3-B01 and S3-B05, while chlorinated solvents were found in S3-B06 and S3-B08. The quantitative data confirmed the screening data.

4.1.2 Dynamic versus Traditional Site Investigation Costs

The rationale for conducting an adaptive sampling and analysis program is to obtain more information about the site while the technical team is in the field. This should lead to faster and better site characterizations at cheaper cost than the traditional approach. At Hanscom Field, VOCs are the primary contaminants that drive the risk assessment and operation of the ground water treatment facility. The dynamic investigation process resulted in 601 soil samples screened and 164 (158 soil and 6 ground water) site and QC samples quantitatively analyzed for VOCs. In addition, 69 and 121 site and QC soil samples were analyzed for PCBs/PAHs and metals, respectively. Soil samples were selectively chosen to test the initial conceptual model for each site in Operable Unit 1. The models were refined in the field as additional data were gathered.

The data produced in the field will be used to evaluate contaminant risk to ground water and what, if any, future action may be taken. HAFB also used the field data (soil, ground and well water) to help optimize VOC ground water collection into the treatment plant. The location of soil contaminated areas adjacent to wells containing high concentrations of VOCs (in some cases pure product) were identified. At Sites 2 and 3, larger capacity pumps were installed during December 1996 in selected wells to increase the flow rate (to 320-gal/min) into the treatment plant. Based on these modifications, influent concentrations increased from 500-ppb in August 1996 to 900-ppb one year later. The ground water recharge system was reactivated in July 1997. Initial checks at some of the wells now show VOC levels as high as 1,200-ppb. It is hoped that influent concentrations will reach levels of 2,000 to 3,000-ppb.²⁵

Comparisons of traditional and dynamic site investigation costs are shown in Table 4. Assume that in the traditional approach, cost estimate 1, the number of samples collected and the types of analyses performed are the same as in the dynamic site investigation. To reach the same end point, consider that the traditional field investigation is conducted in two phases: 1) a screening phase to determine extent and movement of contamination and 2) a more quantitative phase to determine risk to ground water. Off-site laboratory sample analysis costs are based on local laboratory pricing, with data turnaround times typically 2-4 weeks. Higher project costs can occur if samples are analyzed by laboratories with more national reach, presumably due to increased sales and marketing costs and the overhead associated with the wide range of state and federal certification programs (see Table 5).⁵ For phase 1, a total of 661 site and quality control samples are collected over the same 8-day period site samples were collected and screened in the dynamic investigation. Phase 2 begins after the screening data have returned from the laboratory and a new workplan has been prepared. Total project costs, \$142,176, include laboratory analysis sample charges for VOCs, PCBs, PAHs, and metals for site and QC samples as well as sample shipping charges (1,062 sample jars), field team remobilization, and sample collection. The cost for consulting services has been excluded in this analysis.

Table 4. Cost Comparison between Traditional and Dynamic Field Investigations

Estimate 1 Traditional Investigation Off-Site Analysis Data Turnaround 2-4 Weeks		Estimate 2 Traditional Investigation Off-Site Analysis Data Turnaround Two Days		Estimate 3 Dynamic Investigation On-Site Analysis Data Turnaround Next Day	
Predetermined VOC Screening Analysis 601 Site Samples 60 QC Samples	Cost 39,065 3,900	Directed VOC Screening Analysis 601 Site Samples 60 QC Samples	Cost 58,598 5,850	Directed VOC Screening Analysis 601 Site Samples	Cost 19,833
VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 19,750 2,000	VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 29,625 3,000	VOC Quantitative Analysis 158 Site Samples 16 QC Samples	Cost 15,800 1,600
PCB Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 6,800 700	PCB Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 10,200 1,050	PCB and PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 6,800 700
PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 9,860 1,015	PAH Quantitative Analysis 68 Site Samples 7 QC Samples	Cost 14,790 1,523		
Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 36,300 3,600	Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 54,450 5,400	Metals Quantitative Analysis 121 Site Samples 12 QC Samples	Cost 33,275 3,300
Analytical Cost Mobilization Cost Remob to Collect Quant Samples 3 Additional Field Days Sample Shipping Charge	\$122,990 \$5,000 \$5,000 \$6,000 \$3,186	Analytical Cost Mobilization Cost (50% surcharge) 11 Additional Field Days Sample Shipping Charge	\$184,486 \$5,000 \$22,000 \$3,186	Analytical Cost Field Laboratory/Instrument Mobilization Cost	\$81,308 \$10,000
Total Project Cost	\$142,176	Total Project Cost	\$214,672	Total Project Cost	\$91,308

See Table 5 for laboratory and field methods and per analyte group sample analysis cost.

Table 5. Traditional versus Laboratory Sample Charges and Data Turnaround Times

Regional Laboratory	National Laboratory Contract Laboratory Program	Field TDGC/MS Data Turnaround: Quantitative Next Day Screening Same Day	Analyte
Data Turnaround: 14 Calendar Days	Data Turnaround: 35 Calendar Days		
<u>\$125/sample</u> SW 846 method 8260A 35-min/sample analysis <u>\$65/sample</u> SW 846 method 3810 20-min/sample analysis	<u>\$165/sample</u> SW 846 method 8260A 35-min/sample analysis <u>\$65/sample</u> SW 846 method 3810 20-min/sample analysis	<u>\$100/sample</u> modified SW 846 method 8260A 20-min/sample <u>\$33/sample</u> rapid screen TDGC/MS 30-sec/sample analysis	VOCs
<u>\$100/sample</u> SW 846 method 8080 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples	<u>\$150/sample</u> SW 846 method 8080 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples	<u>\$100/sample</u> modified SW 846 method 8270B 10-min per analysis; sample preparation 1-hr/batch of 20 samples	PCBs
<u>\$145/sample</u> SW 846 method 8100/8310 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples	<u>\$255/sample</u> SW 846 method 8100/8310 20-min/sample analysis; sample preparation 2-hr/batch of 20 samples		PAHs
\$300/sample SW 846 method 6010 8-min/sample analysis; sample preparation 2-hr/batch of 20 samples	\$325/sample SW 846 method 6010 8-min/sample analysis; sample preparation 2-hr/batch of 20 samples	\$275/sample modified SW 846 method 6010 8-min/sample analysis; sample preparation 1.5-hr/batch of 20 samples	Metals

Notes: Laboratory costs can vary greatly. The volume of samples analyzed, whether they are received in bulk or over an extended period, the type of QC and documentation required, and the current workload of the laboratory greatly influences the sample analysis charge. Sample charges are based on quotes from commercial laboratories for the analysis of 30 samples delivered in one shipment.

The second cost estimate, \$214,672, is based on expediting the traditional site investigation process. A 50% surcharge is applied to obtain data 24-hours after the off-site laboratory receives the samples. With overnight shipping, data turnaround time is two days. To accomplish the adaptive sampling and analysis investigation in one field mobilization, 21-days will be needed to complete both the screening and quantitative analysis. When compared to the dynamic investigation (10-days), eleven additional days will be needed and has been included in the cost. Using a more conservative sample surcharge of 100% results in total project costs of \$276,166.

Cost estimate 3 is based on the dynamic workplan/adaptive sampling and analysis program. Total project costs, \$91,308, include charges for on-site analysis of VOCs, PCBs, PAHs, and metals, as well as field team, instruments, and laboratory mobilization. The dynamic site investigation takes advantage of the fact that any data surprises will be addressed by decisions made in the field. For instance, no PAH or PCB samples were initially scheduled for analysis at Sites 2 and 3, see Table 6. During VOC screening, it was noticed that the soil was contaminated by petroleum products. Samples from Sites 2 and 3 were subsequently analyzed for PCB/PAH until sufficient data suggested that these contaminants were generally well-below the site-specific action levels.

Total project cost savings between estimates 1 and 3 *and* between 2 and 3 are \$50,868 (36%) *and* \$123,364 (57%), respectively. On the one hand, on-site screening yields the greatest savings as a percent of the total project cost. On the other hand, the analysis of semivolatile organics by TDGC/MS with IFD data analysis produced the greatest per sample cost savings; namely, \$100/sample for PCB/PAH versus \$245/sample. Based on these assumptions, savings are accrued by implementing field analytics when on-site mobilization costs are compared against traditional costs for sample collection, sample shipping charges and the need for multiple mobilizations. It should be reemphasized that these costs exclude all additional consulting costs associated with multiple field investigations.

The IFD data analysis algorithm greatly increases sample analysis productivity when compared against other MS data analysis systems. Unlike standard methods that obtain compound selectivity by adjusting the chromatography to separate organics, selectivity is obtained through the software. IFD reduces the per sample analysis costs in two significant ways. First, the time required to prepare complex environmental samples for analysis is reduced. Second, the time of analysis is reduced by minimizing the reliance on chromatographic separation. In this project, PCBs and PAHs were detected in petroleum contaminated soils in ten minutes without the need for sample cleanup. These productivity gains result in more samples analyzed per day per instrument, while measurement sensitivity was obtained through TD sample introduction. Similarly, the field-practical microwave digestion method reduces sample preparation times over current procedures, while the modification to the ICP optical bench provided stable instrument response under adverse field conditions.

Finally, the on-site adaptive sampling and analysis program can better target areas shown to be contaminated. For example, target compounds were found in 121/158 samples (77%) selected for quantitative analysis. Although 37 samples did not contain measurable contaminants, they were selected to determine the extent of VOC contamination at each site. In this context, several points should be made. First, field analysis produced contaminants in five of fourteen (36%) samples while the off-site confirmatory samples only contained VOCs in two of the fourteen

Table 6. Dynamic Workplan Projected and Actual Number of Samples Analyzed

Type of Analysis	Site 1 Samples		Site 2 Samples		Site 3 Samples		Total Samples Analyzed	
	Projected	Actual	Projected	Actual	Projected	Actual	Projected	Actual
VOC Samples Screened	162	210	135	177	288	214	585	601
VOC Samples Quantified	42	51	36	58	59	49	137	158
PCB/PAH Samples Quantified	42	46	0	12	0	10	42	68
Metals Samples quantified	51	22	44	54	36	45	131	121

Sample number includes field duplicates.

samples (14%). Second, off-site laboratory concentrations were much lower than field results for the two samples where both laboratories did detect VOCs. This VOC loss by off-site laboratories is consistent with findings reported in the literature.^{17,26,27} Third, the adaptive sampling and analysis program can better address the scientific and engineering questions under investigation. For example, HAFB staff have modified the ground water collection system based on the data produced in the investigation. These improvements have increased the VOC influent concentrations into the treatment plant.

4.2 Field Analytical Results

The method data quality objectives listed in Tables 2 and 3 were established to demonstrate that field analytics can support the site-specific HAFB Operable Unit 1 field investigation objectives; namely, to determine future vadose zone soil action (risk-analysis) and to decide whether improvements to the ground water collection system can be made for these sites. Field measurement performance including method selectivity, sensitivity, precision, and accuracy is presented below. Table 7 summarizes these findings, with all supporting data reported in Appendix V.

4.2.1 Evaluation of Screening TDGC/MS Data

The method used in the HAFB dynamic site investigation was developed at Tufts University. The mass spectrometer was operated in the direct measuring mode, with three fragment ions per target compound monitored simultaneously for identification purposes. A total of eleven compounds was screened in less than 30-sec. The TDGC probe head was placed over a small hole made in the Geoprobe™ sleeve at 1-ft intervals. The relative target compound signal heights were recorded at each hole. At the beginning of each day, and when the analyst determined it was needed, a known standard of VOCs was injected into the sleeve to approximate a 10-ppb/compound standard. Samples were not analyzed unless signal responses were ± 2 log units. The direct measuring TDGC/MS, operated in this manner, provided instantaneous and selective detection of each target compound. Whenever a highly contaminated soil was analyzed, the analyst waited until all MS targeted fragment ion signals returned to baseline before analyzing the next sample. This practice insured no carry-over between samples.

Over an eight-day period, 601 individual soil samples were screened at approximately 1-ft intervals. Based on these results, quantitative VOC measurements were made on 144 2-ft composite soil samples. Each 2-ft section of sleeve was cut lengthwise, with soil collected over the tube length and placed into sample collection jars. Samples for quantitative VOC analysis were collected first and were not homogenized. The difference between 158 total soil samples analyzed and the 144 site samples analyzed is due to the number of QC samples. Once the VOC sample had been collected, the remaining sleeve soil was put into a cleaned mixing pan and homogenized for PCB/PAH and metals analysis.

Whenever the screening results indicated the presence or absence of VOCs, quantitative GC/MS confirmed this finding at the measurement reporting limit at a confirmation rate of 90% (142/158 samples). For six of the samples, screening results indicated a positive response while

Table 7. Performance Measurements Obtained in the Field for HAFB

Performance Measurement	VOC Screening by TDGC/MS	Quantitative VOC Analysis by Purge & Trap GC/MS Modified Method 8260A	Quantitative PCB/PAH Analysis by TDGC/MS Modified Method 8270B	Quantitative Metals Analysis by ICP/OES Modified Method 6010
Sensitivity	± 2 log units, daily standard at 10-ppb	3-ppb to 33-ppb, see Table 12 MDL study	total PCBs 200-ppb; PAHs 100-ppb to 400-ppb, see Table 13 MDL study	0.3 to 40-ppm, see Tables 31 and 32 for MDL study
Selectivity	11 target compounds, 3 ions/compound monitored simultaneously	18 target compounds, preselected retention windows and extraction of 3 ions/compound	PCB detection by homologs, 16 target PAHs speciated, preselected retention windows and extraction of 3 ions/compound	22 metals, preselected wavelength for each metal
Precision	± 2 log units, no duplicate analyses performed	average RPD 40 ± 25%, see Table 14	samples selected for replicate analysis contained no measurable concentrations	ERA WastWatR #9967 10% RSD, Table 18; ERA T/CLP Soil #228 3% to 15% RSD, Table 19; site triplicate samples ave RSD 16±15%, Table 22
Accuracy	no sample fortification	surrogate, 83% within DQO, Section 4.2.2; field vs. lab comparison, field measured concentrations greater than off-site analysis, see Table 15	surrogates, 83% within DQO, Section 4.2.2; field vs. lab comparison, only one sample selected for analysis contained reportable concentrations	ERA T/CLP Soil #228 metal dependent average recovery 103±37%, Table 19; field vs laboratory average percent difference 32 ± 15%, Table 21
Other	carryover minimized by on screen baseline monitoring	method blanks made after analysis of high concentration samples	method blanks made after analysis of high concentration samples	method blanks and laboratory control check samples run every 20 th sample

quantitative GC/MS indicated no measurable concentrations at the reporting limit (4% false positive) and the converse (6% false negative) for the remaining ten samples. At the 10-ppb level, the concentration for which the screening TDGC/MS was calibrated, the confirmation rate was 86% (136/158) with 3% false positive and 11% false negative. Finally, if yes/no detection is the benchmark for comparison, a 76% (120/158) confirmation rate is obtained, with 1% false positive and 23% false negative responses. This trend is what should be expected; namely, that quantitative GC/MS is more sensitive than screening MS. Moreover, rapid screening GC/MS measurements were made over a hole the size of a nickel as compared to the composite sampling where samples were collected over a 2-ft section of the sleeve. From the composite sample, a 5-g quantity of soil was analyzed. Comparing measurement accuracy other than by false positives or negatives for discrete and composite soils is nonsensical. It is evident that the rapid screening TDGC/MS method provided an excellent guide to determine where samples should be collected, what VOCs were present, and their relative concentrations in the sample.

4.2.2 Evaluation of Quantitative GC/MS Data

The VOC site maps were produced in the field from data obtained by a standard laboratory purge and trap GC/MS system. The semivolatile data were produced by a Tufts modified TDGC/MS system. All samples were analyzed by full scan mass spectrometry detection, with the data analyzed by the IFD software. To determine sources of deviation, the IFD data generation software was evaluated. Results were consistent with HP's EnviroQuant data analysis software for standard solutions and site samples where the matrix posed little or no data interpretation problems. When high level matrix interferents were present, IFD detected target compounds and internal standards more easily than did EnviroQuant, see Section 5. Based on the results presented below, deviations from the DQO's were in the data generation process, i.e., instrument and human error, and not in the IFD data analysis software.

Initial and Continuing Calibration Comparison Study IFD was used throughout the study to identify and calculate the line parameters of each compound from the total ion current chromatography data. Table 8 lists each VOC's initial 5-point calibration linear regression parameters as well as the average response factor (RFcal) and relative standard deviation (%RSD). The %RSD's were well within the field criterion of $\leq 30\%$ for two-thirds and $\leq 40\%$ for the remaining one-third of the target compounds. VOCs were typically within the DQO's required of fixed-based laboratory data. Shown in Table 9 are the initial and continuing calibration response factor percent differences for the VOCs. The field data meets the data quality objective established for this study with one exception. Vinyl chloride yielded $> 40\%$ differences one-half of the time. It was the only target compound that exists as a gas at ambient temperature. Despite handling the VOC standards with care, we were unable to meet the DQO for this compound in the field.

The DQO's were met for both the initial and continuing PCB/PAH RF calibrations, see Tables 10 and 11. The TD initially installed for semivolatile analysis broke during the first day of the investigation. Carrier gas flow lines developed a leak at one of the solder joints resulting in loss of signal detection. A second TD unit was installed which also developed the same type of leak. Carrier gas line problems were identified by the decreasing internal standard MS signal. This is evident by the poor instrument performance demonstrated during the last RF calibration

of each initial calibration. Nine site samples were not bracketed by an end of day continuing RF calibration verification. Although the TDGC/MS produced lower than normal internal standard responses for six of the nine site samples, none of them showed measurable PAH/PCB signals above the site-specific reporting limits. At the conclusion of the site investigation, the TD was redesigned to minimize manual operation of the sample introduction valve, see Section 5.

Method Detection Limit Study MDLs for VOCs were produced using the purge and trap GC/MS. VOC concentrations were based on an attempt (trade off) to meet the quantitation limit. The lowest concentration where vinyl chloride was observable in the field was 20-ppb. Seven different aliquots (n=7) were prepared and analyzed at this concentration. Table 12 lists the results along with the QL (10DAF) and reporting limit. Each sample measurement, average concentration, standard deviation, and %RSD are shown in the table. All compounds were detected at concentrations below the action level (2 x QL) except for vinyl chloride, methylene chloride, and 1,2-dichloroethane. The poor vinyl chloride measurement precision, 33% RSD, yielded MDL concentrations that exceeded the 5-ppb quantitation limit.

The short timeframe between mobile laboratory setup, the rate of site sample collection, and problems with the TD resulted in the MDL study being performed at the end of the field investigation. By that time, the air in the GC/MS trailer had become contaminated with methylene chloride from the soil/solvent extraction procedure used in the PCB/PAH method. Near constant methylene chloride concentrations were found in the sample data over time. We informed EPA's Office of Environmental Measurement and Evaluation (OEME) staff that the methylene chloride data had become compromised. After discussion, it was agreed that methylene chloride should be taken out of the target compound list.

One lesson learned is that better laboratory ventilation is needed for future field work to reduce solvent contamination of the mobile laboratory from the sample preparation procedure. Another lesson learned is that obtaining such low limits of detection for the VOC gases in the field may not be achievable. Therefore, it is better to insure that the DQO's are met for all other VOCs rather than compromising on data quality. For example, conducting the MDL study at 10-ppb would have resulted in meeting the QL for 1,2-dichloroethane. Visual inspection of the 1,2-dichloroethane peak signal showed that under the experimental conditions employed, lower measured concentrations could have been achieved.

For PAH and PCBs, the MDL study was conducted prior to the initial calibration. MDL's were determined by taking 2-g of ERA soil previously analyzed and shown to contain no detectable organics. The soil was added to a 7-ml vial and fortified to contain 300-ppb of each PAH and 300-ppb of Aroclor 1248. The soil was extracted by hand-shaking with 2-ml methylene chloride. The soil/extract was centrifuged for 3-min and the extract placed into a 2-ml sealed vial. Seven soil samples were extracted and analyzed by TDGC/MS. Table 13 lists the peak areas, average area, standard deviation, and %RSD as well as the MDL and QL.

Benzo(a)pyrene and dibenzo(a,h)anthracene have the lowest PAH action level (20DAF) concentrations, viz., 2-ppm each. All other PAH 20DAF concentrations are \geq 5-ppm, with quantitation limits \geq 2.5-ppm. The site-specific QL for total PCBs was set at 0.5-ppm. The measured MDL's for PAH were between 87-ppb and 374-ppb. The MDL for total PCB was 153-ppb. TDGC/MS measurements easily met the MDL DQO's for this project. Moreover, PCB and

PAH were detected in the presence of each other in one 10-min analysis, with measured MDL concentrations below typical laboratory detection limits. Standard laboratory methods generally require two different analyses; GC/MS for PAHs (method 8270B) and GC with electron capture detection (ECD) for PCBs (method 8080A). The estimated quantitation limit for these methods with gel permeation chromatography cleanup of soil extracts is ~ 660-ppb in soil. Because the TD can introduce volumes of up to 500-uL of extract, standard syringe injection of 1 to 2-uL volumes require 10-g of sample compared to the 2-grams used in this method.

The IFD software allows the mass spectrometer to operate as a universal detector and, at the same time, provide selective data analysis capability. Although MS instruments can provide increased sensitivity when operated in the selective ion monitoring (SIM) mode, the universality of full total ion current mass spectrometry is lost when library matching (compound identification) of nontargeted compounds is needed. Sample concentrations were reported and flagged (J) whenever the measured concentrations were between the MDL and RL (the low calibration standard).

Measurement Precision The sampling and analysis plan required field duplicate (FD) samples to be collected every twentieth sample. Seven samples were collected and analyzed as field duplicates. These samples were obtained by taking soil from the same 2-ft section of sleeve as the initial site sample. One site sample was analyzed as a triplicate. Initial and field duplicate samples were not typically analyzed on the same day. The goal was to analyze as many unique site samples as quickly as possible to confirm the screening results and to direct the final round of sample collection. Measurement precision results are shown in Table 14. Field replicate samples were run either as duplicates or triplicates. All positive responses are shown. (Benzene produced low level background signals at the MDL.) Relative percent differences (RPD) and RSD are reported when multiple samples from the same container were analyzed two or three times, respectively.

Unfortunately, all but two samples analyzed multiple times contained compounds at trace levels. The DQO measurement precision was $< 5 \times \text{QL}$, $\text{RPD} < 100\%$ and at $> 5 \times \text{QL}$, $\text{RPD} < 60\%$, see Table 2. For samples where target compounds were found in all replicates, the measurement precision DQO was met in every case. In four instances where measurable quantities were obtained in one replicate but not the other, concentrations were well below each target compound's quantitation limit. These results are consistent with past data where concentration levels are at the detection limit.

Measurement precision is highly dependent on the variance in sample homogeneity. Screening data showed that there can be great differences in concentration from one foot to the next within the same 4-ft sleeve. Since initial and field duplicate samples are collected by scooping soil over a 2-ft section, the differences in the initial and field duplicate VOC concentrations may be attributable to sampling and the loss of analyte by the time FD samples were analyzed. Another lesson learned was that preselecting the samples for determining measurement precision and accuracy falls in much the same category as prespecifying the samples to be collected in the traditional investigation process, i.e., many data are generated with little information value produced.

Measurement Accuracy During the planning session it was agreed that matrix spike experiments were not needed as part of the site-specific DQO's. Surrogates were added to VOC and semivolatile site samples. Known and measured surrogate concentrations were compared. For VOCs, 158 site samples were analyzed. The average surrogate recovery, either 4-bromofluorobenzene or 1,4-difluorobenzene, was $132 \pm 44\%$, with 82% of the analyses within the data quality objective, see Table 2. After data review, it appears that 4-bromofluorobenzene yielded recoveries ($87 \pm 40\%$) closer to the fortified value than did 1,4-difluorobenzene. The 4-bromofluorobenzene measured concentration was within the DQO 92% of the time. The average semivolatile surrogate (octachloronaphthalene) recovery was $94 \pm 49\%$, with 92% of the analysis within the data quality objective. The mass range limitation of the HP GCD mass spectrometer to 425 amu resulted in the selection of secondary ions used to quantitate octachloronaphthalene resulting in poor signal sensitivity.

Field versus Laboratory Comparison Study The dynamic workplan for Operable Unit 1 called for the fifth sample and every tenth subsequent sample to be sent off-site for laboratory analysis. A total of fourteen samples was analyzed by the two laboratories for VOCs and five samples for PCB/PAH. Analysis of the samples listed below produced no reportable VOC levels for the target compounds by both laboratories: S1-B2-(2-4), S1-B8-(6-8), S1-B17-2-(10-12); S2-B6-(2-4), S2-B10-(10-12), S2-B12-(12-14), S2-B15A-(10-12); S3-B6-(10-12), S3-B14-(6-8). Field and laboratory results where one of the laboratories obtained reportable data for VOCs are listed in Table 15.

In no instance did the off-site laboratory find target compounds where field analysis did not. For samples where either the matrix (sample interferences) or target compounds were low in concentration, i.e., no dilution of sample was required, field analysis found low levels of VOCs where the off-site laboratory did not. It has been well documented in the literature that VOCs are lost in transport and/or storage by the time off-site laboratories analyze the sample.^{25, 26}

In addition to potential VOC losses, samples S2-B2-(20-22) and S3-B1-(13-15) required 5:1 and 50:1 dilutions, respectively, before analysis by the off-site laboratory. The IFD software minimizes the masking of low concentration target compounds by high concentrations of matrix interferences and/or other target compounds. The data analysis software can "look-through" non target ion signals and determine compound identity unambiguously. Field analyzed samples were only diluted when target compounds overloaded the MS detector. The project objectives allowed for samples to be quantitated outside the initial calibration range for the field analyses. This is best illustrated by the field and laboratory results for S2-B2-(20-22) where 1,1-dichloroethene and 1,1-dichloroethane were diluted by the laboratory below the MDL and where cis-1,2-dichloroethene was masked by a matrix interferant. Although there is very little positive response data to compare, whenever field analysis produced a positive identification at high target compound concentrations so too did the off-site laboratory.

4.2.3 Metals Analysis by ICP/OES

ICP/OES Initial and Continuing Calibration Comparison Study ICP/OES instrument manufacturers typically require laboratory environments of $\pm 5^\circ\text{C}$ to insure instrument performance. This is sometimes difficult to achieve in the field. Therefore, a site-specific

instrument performance DQO of $\pm 20\%$ was established. Although lead and cadmium were the target analytes of concern for the HAFB field investigation, initial calibration plots were made on a daily basis for 22 inorganics. Tables 16 and 17 show the average percent recoveries and their %RSD's obtained over a 7-day period for both the initial and continuing calibrations, respectively. The average percent recoveries for both the initial and continuing calibrations were well-within $\pm 20\%$ for all elements including silver and antimony. The 25% 3:2 HNO₃:HCl mixture produces a more stable environment for these two metals than the standard EPA method using 5-10% HNO₃ solution.

Measurement Accuracy Table 18 lists the ICP/OES limit of quantitation (LOQ), certified value, and percent recoveries (%R) for an ERA, WasteWatR #9967, laboratory control check standard. This sample was analyzed at the beginning and end of each day. The MDL for lead (5-ppm) and cadmium (0.33-ppm) is the LOQ x 43.28, i.e., the weight factor of the acid digestion solution. The percent recovery data for this sample are shown in Table 18. The average percent recovery (%R) between the measured and certified values was within $\pm 20\%$, with the %RSD generally $< 10\%$. A second laboratory control check sample, PriorityPollutnT/CLP Soil # 228 also purchased from ERA, was analyzed every 20th sample. The average percent recovery for nine measurements over a 7-day period is shown in Table 19. Measurement precision was excellent with recoveries closer to the made-to concentrations than the certified value. The probable difference in accuracy is in the extraction procedure used to determine the certified value. EPA method 3050 is an open hot plate acid digestion procedure as opposed to the field microwave digestion method, see Section 5.

Matrix Interferant Test Solution A standard solution (A) containing calcium, magnesium, aluminum, and iron was used as the interferant solution. A second solution (Sol AB) containing these inorganics as well as twelve other constituents were analyzed. The average percent recoveries for the combined standard were within $\pm 10\%$ of the certified value, see Table 20.

Field versus Laboratory Results Comparison of field versus laboratory data for lead and cadmium has been tabulated in Table 21. The percent difference was calculated by dividing the difference between the field and laboratory concentrations by the mean value of the two. Excellent agreement was found between the field and laboratory results. Soil from sample S2-B2-(20-22) was prepared for analysis three times and found to contain lead at 16-ppm $\pm 4\%$ RSD. Replicate analysis for this sample was consistent with measurement precision results produced at the conclusion of the HAFB investigation, see Section 5.

Measurement Precision In addition to S2-B2-(20-22), nine other samples collected from the site were prepared and analyzed three times. These samples were selected randomly from each batch of twenty samples. Table 22 lists the results for cadmium and lead as well as the other inorganics. In general, the average concentration %RSD's were less than 30% for each element except for sample S2-B4-(19-21), which produced values as high as 70% for six of the seventeen inorganics present in the sample. These results are remarkable given the heterogeneous nature of the soil and the high degree of petroleum contamination in some samples; 128 of the 135 detectable responses (95%) produced RSD's $< 30\%$.

4.2.4 Summary of Findings: Field Analytical Instrument Performance

The data produced in the field met the DQO's established for this project. Where site geology is amenable, the Geoprobe™ can collect soil samples much faster than the time required to screen and process each 4-ft sleeve, including:

- tube preparation and TDGC/MS screening time
- the preparation of samples for subsequent quantitative field and off-site (laboratory) analysis for VOCs, PCBs, PAHs, and metals
- cleanup of the work area prior to analyzing the next tube
- completion of chain-of-custody forms, and
- geological logging of soil sample.

Nonetheless, one analyst processed 75 samples per day. The total cycle time for each 4-ft sleeve was approximately 20-min. Focusing only on sample screening analysis rates without careful planning and on-site decision making can lead to over collection of samples and the loss of VOCs before quantitative measurements can be made. Based on the chain-of-custody, 66% of the sleeves were screened for VOCs the same day they were collected, with the remaining sleeves analyzed within a 24-hr period. Samples were selected for quantitative VOC analysis on a daily basis to delineate contamination based on the screening data. All soil samples were quantitatively analyzed within the 14-day holding period. Most samples were analyzed within 5-days of collection or in the first week of the investigation. These were generally pit and surrounding samples. Longer holding time samples were typically collected from the boundary of the contaminated areas. These samples contained no measurable VOCs as confirmed by quantitative GC/MS at reporting level concentrations.

<u>From Date of collection</u>	<u>Cumulative Daily Analysis</u>	<u>From Date of Collection</u>	<u>Cumulative Daily Analysis</u>
same day	14%		
1	35%	6	87%
2	48%	7	91%
3	59%	8	96%
4	74%	9	98%
5	82%	10	100%

PCB, PAH, and metal samples were also analyzed within sample holding times. The combination of TD sample introduction and the mass spectral algorithms developed for both the Bruker and HP mass spectrometers provided the necessary sensitivity and selectivity for compound speciation under fast GC operating conditions. The modification made to the ICP/OES resulted in a stable instrument capable of providing quantitative data in the field. The data produced in this project was in conformance with the site-specific sensitivity, precision, and accuracy DQO's.

Although enzyme immunoassay kits were originally proposed, field analysis found PAH in only six samples and PCBs in only three samples. All sample concentrations were below typical enzyme kit detection limits. The original proposal also included in situ organics detection using a cone penetrometer. Studies performed by Applied Research Associates (ARA), Fugro, and CFAST staff showed that the HAFB site was not amenable to cone penetrometer detection. For example, ARA snapped the cone tip and bent the pipe in an attempt to collect ground water during an Air Force/Tufts University demonstration at HAFB.

Table 23. Screening VOC Analysis of Soil Samples

QC Parameters	Field Method TDGC/MS Screening Analysis	Field Method Heated Headspace GC Screening Analysis Method 3810
Sensitivity	±2 log units daily standard at 10 ppb	1 ppm standard adjust to > 2 times background
Selectivity	48 ions can be monitored simultaneously, 16 target compounds can be preselected if 3 ions/compound are monitored	adjust chromatography to separate VOCs of interest
Precision	no duplicate analyses performed	no data available
Accuracy	no fortified analyses performed	no data available
Other	carryover minimized by on screen baseline monitoring	carryover monitored by analysis of blanks, watch baseline on chromatograms

5.0 Technology Transfer

Over the past decade, research has led to field-practical TDGC/MS methods of analysis for organic compounds. In addition, field-practical sample preparation procedures for the digestion of metals from soils have also been developed, with modifications made to the ICP/OES instrument for field application. Tables 23-26 were developed as an outgrowth of this project to illustrate current SW 846 QC requirements for volatile and semivolatile organics and metals analysis and their corresponding field methods. The purpose of these tables is to show that field analytical technologies can produce data quality equal to standard laboratory instrumentation and methods.

Table 24. VOC Analysis of Soil Samples by Purge and Trap GC/MS

QC Parameters	Field Method SW-846 Modified Method 8260A	Laboratory Analysis SW-846 Method 8260A
Instrument Performance Tests for MS Tuning	perform check as per instrumental method, minimum requirement once to initiate shift	perform check as per instrumental method, minimum requirement once to initiate 12-hr shift
Initial Calibration 5-point	DQO dependent; can match SW 846 or all RF %RSDs $\leq 40\%$ and no more than $\frac{1}{3} > 30\%$ or all RF %RSDs $\leq 30\%$	calibration check compounds (CCC) %RSD's must be $< 30\%$, if all RF %RSD $\leq 15\%$ then use Ave. RF else use linear regression
Laboratory Control Standard	sample throughput dependent, can match SW 846	after each initial calibration; percent accuracy within 80% to 120%
Continuing Calibration Verification	DQO dependent; can match SW 846 or begin & end of day, percent difference for all compounds $\leq 40\%$ and no more than $\frac{1}{3} > 30\%$	one per 12-hr shift; (calibration check compound) CCCs $< 20\%$. All analytes within $\pm 25\%$ of expected value
Method Blank	once per day and after highly contaminated sample; all target compound concentrations $< \text{PQL}$	one per analytical batch; all target compound concentrations $< \text{PQL}$
Surrogate Spike Analysis	DQO sample throughput dependent; for each sample, blank, standard or other QC run	for each sample, blank, standard or other QC run, laboratory established recovery limits (e.g. 80-130 %)
Sensitivity	5-2500 ppb levels, matrix dependent	5-2500 ppb levels, matrix dependent
Selectivity	can do up to 97 VOCs with 2-6 ions per compound; minimal chromatographic separation employed, selectivity is achieved by data analysis algorithms	can do up to 97 VOCs with 1-6 ions per compound; adjust chromatography to separate VOCs of interest
Precision	replicate analysis QC acceptance criteria	replicate analysis QC acceptance criteria
Accuracy	sample throughput dependent; can match SW 846; laboratory control check sample (LCS) once per day	surrogate dependent recovery within 70-120%; laboratory control check sample (LCS) once per 12-hr shift
Other	carryover monitored by analysis of blanks, watch baseline on chromatograms	carryover monitored by analysis of blanks, watch baseline on chromatograms

Table 25. SVOC Analysis of Soil Samples by Thermal Desorption GC/MS

QC Parameters	Field Method SW-846 Modified Method 8270A	Laboratory Method SW-846 Method 8270B
Instrument Performance Tests for MS Tuning	perform check as per instrumental method, minimum requirement once to initiate shift	perform check as per instrumental method, minimum requirement once to initiate 12-hr shift
Initial Calibration 5-point	DQO dependent; can match SW 846 or all RF %RSDs \leq 40% and no more than 1/3 > 30%	calibration check compounds (CCC) %RSD's must be < 30%, if all RF %RSD \leq 15% then use Ave. RF else use linear regression
Laboratory Control Standard	DQO sample throughput dependent; after each initial calibration, percent accuracy within 80% to 120%	after each initial calibration; percent accuracy within 80% to 120%
Continuing Calibration Verification	DQO dependent; can match SW 846 or begin & end of day, % difference for all compounds \leq 40% and no more than 1/3 > 30%	one per 12-hr shift; %D for all compounds \leq 20%
Method Blank	once per extraction batch; all target compound concentrations < PQL	one per extraction batch; all target compound concentrations < PQL
Surrogate Spike Analysis	sample throughput dependent; for each sample, blank, standard or other QC run	for each sample, blank, standard or other QC run, laboratory established recovery limits (e.g. 20-130 %)
Sensitivity	100-ppb to 1000-ppb	660-ppb to 3300-ppb
Selectivity	can do up to 350 SVOC with 2-6 ions per compound; minimal chromatographic separation employed, selectivity is achieved by data analysis algorithms	can do up to 350 SVOC with 2-5 ions per compound; adjust chromatography to separate SVOC of interest
Precision	replicate analysis QC acceptance criteria	replicate analysis QC acceptance criteria
Accuracy	sample throughput dependent; can match SW 846 for surrogate and MS/MSD recoveries	surrogate recovery compound dependent; MS/MSD per extraction batch
Other	carryover monitored by analysis of blanks, watch baseline on chromatograms	carryover monitored by analysis of blanks, watch baseline on chromatograms

Table 26. Metals Analysis of Soil Samples by ICP/OES

QC Parameter	Field Method SW 846 Modified Method 6010	Laboratory Method Superfund Hazardous Waste Analysis CLP SOW ILMO1.0
Initial Calibration	1 standard and a blank, daily, or every 24-hrs	1 standard and a blank, daily, or every 24-hrs
Calibration Verification	mid-range standard begin, end, and every 10 samples; recovery 80-120%	mid-range standard begin, end, and every 10 samples, or every 2-hrs; recovery 90-110%
Interference Check Sample	beginning of each run; recovery 80-120%	beginning and end of each run or 2 every 8-hrs; recovery 80-120%
Calibration Blanks	begin, end, and every 10 samples; all analytes \leq LOQ	begin, end, and every 10 samples or every 2-hrs; all analytes \leq CRDL
Preparation Blank	1 per SDG or digestion batch; all analytes \leq LOQ except Fe & Al	1 per SDG or digestion batch; all analytes $<$ CRDL
Laboratory Control Standard	Beginning and end of each run; recovery 80-120%	1 per SDG, digestion batch, or matrix; recovery 80-120%
Duplicate Samples	1 per SDG or every 20 samples; $<30\%$ RPD for values \geq LOQ flag value if out	5% or 1 per SDG per matrix per level ; $<20\%$ RPD for values $\geq 5 \times$ CRDL $\pm 1 \times$ CRDL for values $< 5 \times$ CRDL
Sensitivity	100-ppb to 10,000-ppm levels, matrix dependent	100-ppb to 10,000-ppm levels, matrix dependent
Selectivity	22 elements spectrally resolved with no need for interelement correction factors	22 elements mostly resolved with provisions for interelement correction (instrument dependent)
Precision	triplicate analysis 1 per SDG or every 20 samples; RSD $< 30\%$	replicate analysis QC acceptance
Accuracy	soil and standard 1 per SDG or every 20 samples; recovery 70-130% except Al, Fe, Na	no data available
Other	carryover monitored by sequential scans of rinse solution; rinse time between samples 3-min	no data available

5.1 Organics Analysis

Field methods have evolved from an instrument specifically designed for chemical warfare detection (Bruker Instruments) to more typical instruments found in the laboratory (Hewlett Packard). The Bruker GC/MS weighs about 400-lbs and has a footprint of 2-ft x 2-ft x 3-ft. It is field-rugged and was used in the Gulf war. In contrast, typical laboratory instruments are about one-half the size and weigh approximately 50-lbs. Two key technologies provide the core breakthrough for fast GC/MS analysis. First is the mass spectral data analysis software. The U.S. Patent Office (April 1997) issued a Notice of Allowance for all claims related to the mass spectral data interpretation patent. The software provides the unique capability of extracting between two and ten characteristic fragment ions produced in mass spectrometry from targeted organic compounds. Based on a set of mathematical algorithms, compound identity and concentration are determined. Although all MS systems provide MS ion extraction, they cannot determine compound presence using current statistical or library matching routines. The Tufts approach provides compound identification in complex environmental samples without the need for extensive sample cleanup. It is hoped that the underlying mathematical algorithms and process are amenable to other analytical instruments that produce characteristic signatures for targeted analytes like optical emission spectroscopy.

The second technology breakthrough is the thermal desorber (patent pending). Although we had some plumbing (gas leak) problems in the field during the investigation, these problems were fixed and the TD capability was demonstrated. Unlike other commercially available units, it can be ballistically heated from subambient temperatures to 320 °C in 8-sec. The TD uses a standard Tenax tube for VOCs. An empty glass tube is used for direct desorption of organics from solid materials or organic extracts. The latter sample introduction method was used in the HAFB field investigation for PCBs and PAHs.

To test the data generation software, comparisons were made between the EnviroQuant and IFD software systems. Also shown in Tables 8-11 are the initial and continuing calibration line parameters and %RSD RFcal and the percent difference as determined by the EnviroQuant software. Note that all GC/MS total ion current chromatograms were acquired by HP's data acquisition system. The same data files, therefore, were analyzed by both IFD and EnviroQuant. The tables reveal no statistical differences in the information obtained. Moreover, the %RSD's for the eighteen VOC RFcal's were well within the field criterion of $\leq 30\%$ for two-thirds of the target compounds and $\leq 40\%$ for the remaining one-third of the organics established for this field study. Good agreement was also found for PCBs and PAH except for naphthalene. EnviroQuant produced a 65% RSD RFcal in comparison to the 37% RSD calculated by IFD. Excellent agreement was also obtained between the two data analysis systems for the continuing calibration data. The VOC and semivolatile data were typically within the 30% required of fixed-based laboratory data.

To determine instrument and method stability in the field, each sample's internal standard signal was compared against the corresponding average internal standard signal calculated over the first two calibration curves. The internal standard signal was inside the -50 to 150 percent difference HAFB DQO for 134 of 157 samples (85% success rate) analyzed by IFD. EnviroQuant fell outside of the DQO for 32 of the same samples, while missing the internal standard identity in two samples completely for a 78% success rate. Both data analysis systems fell out of QC because of coeluting matrix constituent(s) that affected the internal standard integration.

Table 27 shows IFD and EnviroQuant site sample data comparisons for the same calibration files where at least one of the data analysis systems identified a target compound above the reporting limit. Differences between the two calculated concentrations were due to each data system's peak identification and integration routines. For example, IFD provided better integration where matrix constituents coeluted with either target compounds or internal standards. EnviroQuant cut peaks in half depending upon where peak separation was determined by the software. Target compound concentrations in file #'s 100.D, 178.d, 179.D, and 194.D were reported by IFD whereas, EnviroQuant estimated their concentration below the reporting limit. In addition, EnviroQuant missed compounds in file #'s 102.D, 143.D, and 178.D that were well above the reporting limit, with IFD missing one compound in file # 031.D. Of the 205 reported compounds by both software systems, 85% had RPD's $\leq 50\%$ and 65% had RPD's $\leq 20\%$. This is remarkable given that both data systems identified peaks and integrated signals very differently.

The TD is temperature controlled to minimize introduction of matrix interferences into the GC/MS. It must be continually purged by an inert carrier gas such as helium to exclude oxygen from entering the GC/MS. The purpose is to minimize GC stationary phase degradation at elevated temperatures and to avoid filament burnout in the ionization chamber of the MS. Because of problems with the TD/manual injection valve construction (leaks), an electronically controlled and ballistically heated (ambient to 320 °C in 8-sec) TD unit was built at the conclusion of the HAFB field investigation. Reducing manual manipulation of the TD unit should result in less strain on the TD carrier gas flow lines. An evaluation of the purge and trap TD sample introduction system was made for VOCs. It included a Supelco 15-ml sparging vessel connected on one end to an inert gas supply and on the other end to a Tenax tube supplied by SKC, Inc. (Canonsburg, PA). A 5-min purge time for VOCs was used. The Tenax tube was placed in the TD, with VOCs desorbed onto the GC column. Table 28 shows the initial calibration results for the standard laboratory Tekmar and Tufts systems. The calculated RFcal %RSD's were well below the DQO criteria. Table 29 shows the MDL results. The MDLs for all target compounds were below the quantitation limit, note that the lowest standard was 6.7-ppb as compared to 20-ppb in the HAFB field investigation.

Tekmar and Tufts purge and trap data comparison shown in Table 30 differed in the purge conditions employed and in the GC operating temperature. The same MS was used to acquire VOC data. HAFB site samples initially contained a wide range of target compound concentrations when analyzed in the field. Evident from these results was the dramatic loss of VOCs from the sample. RPDs were within the DQO's when both measurement techniques found contaminants. Consistent with the MDL study, it appears that the Tufts purge and trap/TD can provide lower measurement detection for VOCs conspicuous by the twelve detectable contaminants found by Tufts as opposed to Tekmar.

5.2 Metals Analysis

Table 31 lists the ICP/OES instrument detection limit study for the Target Analyte Metals (excluding mercury). Individual standards were prepared and analyzed seven times according to standard EPA procedures. Table 32 compares the calculated MDL and 10DAF concentrations for these metals. Note that the field instrument can achieve lower MDL's than required for the Hanscom project.

Table 16 lists the daily recoveries for the target analyte metals that can be measured by ICP/OES, while Table 17 shows the daily continuing calibration data. Percent recoveries were excellent for all metals, with 9% the largest deviation from 100% recovery. Table 19 illustrates the percent recovery for a standard reference soil (ERA Priority/PollutnT/CLP). The ERA sample, Soil #228, was analyzed every 20th sample. A total of nine samples was analyzed producing average percent recoveries much greater than 100% when compared against the certified value. The certified value represents the concentration obtained if the soil sample is prepared for analysis by EPA method 3051. The extraction procedure employed a 50% 3:2 HNO₃:HCl as opposed to concentrated HNO₃. Comparison of the data reveals that the average percent recoveries fall much closer to the theoretical (made-to) concentrations as prepared by ERA than they do to the certified values. The results show that greater recoveries are obtained for Na, Ag, Al, Fe, and Sb employing 50% 3:2 HNO₃:HCl as the digestate than in the standard EPA microwave digestion method. Importantly, the stability of all the metals in a single acid mixture (6-months) reduces the number of sample preparation steps to reconstitute antimony and silver from the standard EPA acid digestion solution (stability < 1-day).

Replicate analysis of the site samples shown below produced data consistent with measurement precision results found in the HAFB investigation when experiments were performed to evaluate the performance of the field sample preparation method (50% 3:2 HNO₃:HCl) and standardized EPA method 3501 (50% HNO₃).

	50% 3:2 HNO ₃ :HCl		50% HNO ₃		Number of
	<u>Ave Pb (mg/kg)</u>	<u>% RSD</u>	<u>Ave Pb (mg/kg)</u>	<u>%RSD</u>	<u>Digestions</u>
S2-B06-(2-4)	41	7	39	27	3
S2-B01-(16-18)	181	12	153	11	9
S3-B01-(13-15)	61	19	43	24	3
S3-B06-(10-12)	20	10	18	14	6

The two different microwave extraction procedures produced comparable data for lead. Recall that for S2-B06-(2-4), the commercial laboratory produced a concentration of 168-mg/kg as compared to 46-mg/kg (n=1). The off-site laboratory prepared soil samples by EPA Method 3050. In this method, the sample is refluxed in concentrated nitric acid and hydrochloric acid in an open vessel. It is a total digestion method that can produce greater analyte matrix dependent recovery for some metals over others. For lead, field versus laboratory percent differences were < 60%. Nonetheless, much debate exists in the literature over the benefits of microwave versus open vessel digestion.^{28,29}

X-ray Fluorescence (XRF) and ICP/OES Data Comparison Study At the conclusion of the field investigation, the Spectrace tabletop energy dispersive XRF was evaluated. Soil samples from the site were homogenized and directly placed into the XRF sample cup. Each sample was analyzed three times for lead. EDXRF results are compared against field and laboratory ICP/OES data for four samples.

	XRF (mg/kg) Ave Pb (%RSD)	field ICP/OES Pb (mg/kg)	off-site Lab Pb (mg/kg)	Total Ave Pb (%RSD)
S2-B01-(16-18)	128 (12)	194	222	181 (27)
S2-B06-(2-4)	39 (13)	46	168	84 (86)
S3-B01-(13-15)	57 (13)	65	76	66 (14)
S3-B06-(10-12)	24 (13)	19	29	24 (21)

Excellent measurement precision was obtained by the Spectrace instrument. In addition, the intermethod measurement accuracy was good for all samples except S2-B06-(2-4). Evident from the XRF and ICP/OES replicate analysis data is that the true lead concentration in the sample lies closer to 40-ppm than 170-ppm. The plot of EDXRF and ICP/OES data for 53 site samples is shown in Figure 10. Good correlation was obtained, with somewhat higher concentrations measured by EDXRF; $r^2 = 0.95$, slope 0.96 ± 0.03 and intercept 11 ± 2 . Only twelve site samples contained cadmium. The correlation of EDXRF with ICP/OES data was much poorer; $r^2 = 0.81$, slope 3.1 ± 0.5 and intercept -3 ± 8 . The Spectrace EDXRF with 200 livetime seconds can produce quantitative data in the field for lead and useful screening quality data for cadmium. At 120 livetime seconds the instrument continues to produce quantitative data for lead and screening data for cadmium. The shorter analysis time per element results in higher sample throughput rates. A 10-sample batch can be analyzed in 40-min for lead and cadmium. When choosing a field method, the microwave extraction and ICP/OES can quantitatively analyze a batch of ten samples for Target Analyte List metals except mercury in 125-min.

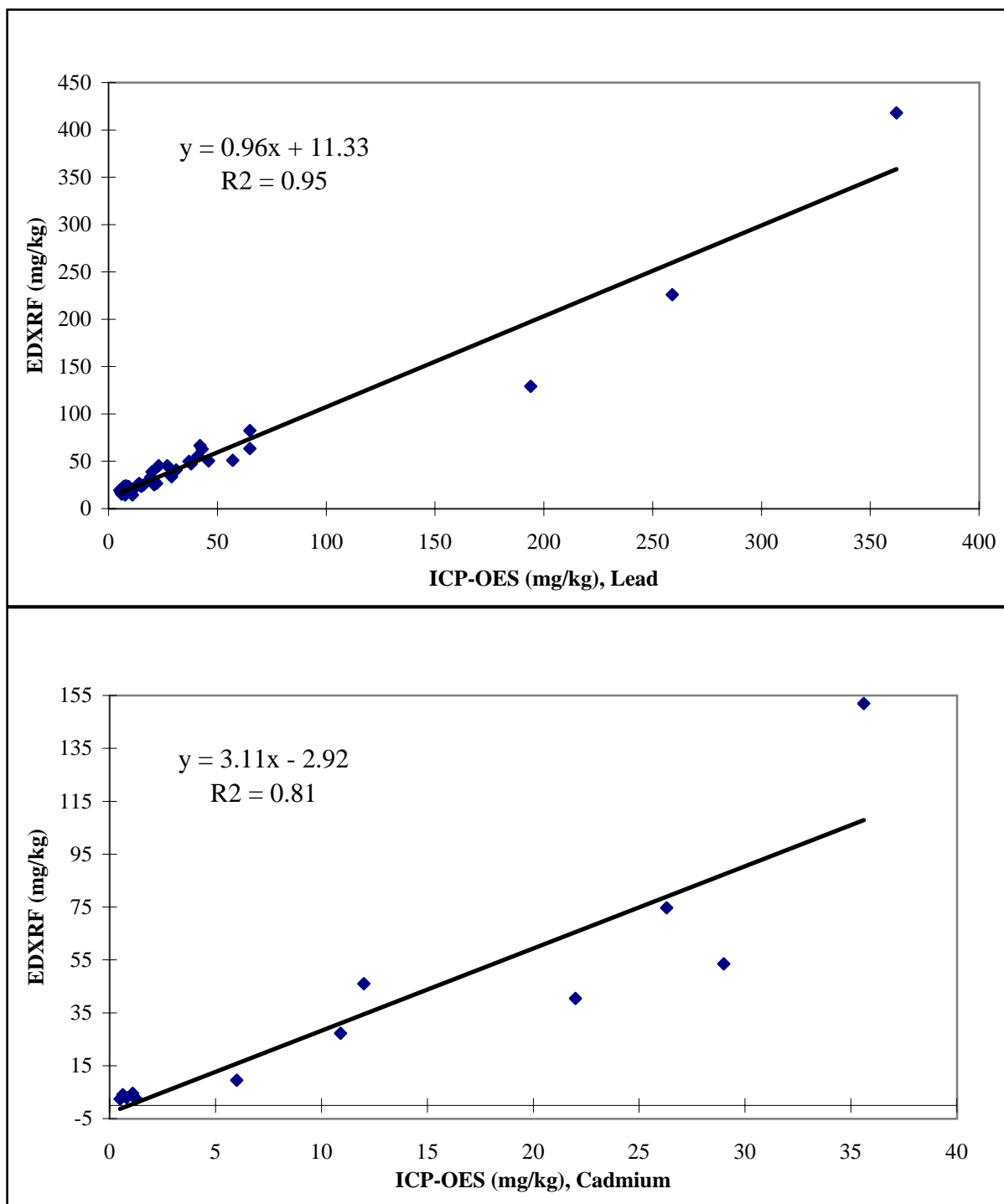


Figure 10 Plot of ICP/OES vs EDXRF for Lead and Cadmium

5.3 Lessons Learned

Summarized below are key findings learned while conducting the HAFB investigation.

Dynamic Workplans and Field Analytics

1. Field analytics employing performance-based methods can produce data of equal quality to fixed-based laboratories employing standardized EPA methods.
2. Field analytics can support a dynamic workplan/adaptive sampling and analysis program.
3. Field analytics can support risk assessments and cleanup verification programs.
4. Cost effectiveness is maximized when site DQO's, analytical throughput rates, data turnaround times, sample collection rates, and sample analysis costs are evaluated and optimized to meet the site-specific scientific and engineering questions under investigation prior to the beginning of the field work.
5. The technical team should be in daily communication, with one person assigned the responsibility for making all final field decisions.
6. Field analytics can result in a higher percentage of samples collected and analyzed containing target compounds when an adaptive sampling and analysis program is implemented compared to the phase engineering approach.
7. Preselecting samples to determine measurement precision and accuracy as in the traditional workplan concept provides minimum information value. The field team should select 5% to 10% of the site samples analyzed in the field. Based on actual results, 70% of these samples should contain target compounds. The remainder should be selected to insure that the boundary of contamination has been established.
8. VOC loss is less when field analytics is used to support the field investigation or cleanup verification program. The longer the holding time and lower the VOC concentration, the more accentuated the difference between on and off-site results.

Field Instrument and Method Performance

9. TDGC/MS and the mass spectrometry data analysis algorithms allow more samples to be analyzed per day than current MS vendor data analysis systems, probabilistic library sample identification matching routines, forward/backward regression search routines, or compound identification through the standard EPA/NIST library matching data systems.
10. The software algorithms can be used to obtain compound selectivity rather than adjusting the gas chromatography operating conditions. This decreases the per sample analysis time and increases the number of samples that can be analyzed per day per instrument over standard GC/MS instruments.

11. Software algorithms can “look through” non target MS ion signals and unambiguously determine compound identity, minimizing masking of low concentration target compounds by high concentration matrix interferents.
12. In this context, low level target compounds are not lost because of the need to dilute sample. Software makes sample dilution less necessary.
13. TDGC/MS provides increased method detection limits over standard syringe sample introduction techniques for GC/MS and comparable detection limits with GC/ECD without the need for a sample preconcentration step.
14. TDGC/MS and the mass spectrometry data analysis algorithms allow PCB/PAH analyses to be performed at the same time without the need for sample cleanup and fractionation time.
15. DQO’s can be met for all target compounds except vinyl chloride. A trade-off may need to be considered between achieving low limits of detection for VOC gases and meeting DQO’s for all other VOC target compounds.
16. ICP/OES modifications resulted in a stable instrument during field operation.
17. A 25% 3:2 HNO₃:HCL mixture produced a more stable environment for the digestion of all Target Analyte Metals with the exception of mercury for quantitative ICP/OES analysis.
18. A 25% 3:2 HNO₃:HCL produced a stable acid mixture (6-months) and high recoveries for silver and antimony as compared to the standard EPA digestion methods (1-2-days).
19. Microwave digestion is field-practical when compared to open vessel acid digestion, with recoveries comparable to what can be obtained in the laboratory.
20. The microwave digestion procedure reduces the number of sample preparation steps and thus time, compared to the standard EPA microwave digestion method.
21. Increased XRF sensitivity can be achieved with an x-ray tube source as opposed to radionuclide sources.

Mobile Laboratory Set-up and Operation

22. A minimum of one week is required to install and calibrate all field instruments. MDL study should be performed prior to beginning field work.
23. Depending on the number of field instruments, separate electrical services should be provided per instrument compliment.
24. Line voltage regulators are recommended to protect instruments and computers from line voltage surges or brownouts.

25. Instrument backup or service repair plan should be incorporated into the workplan. For example, the TD carrier gas leakage problem was addressed by using the Tekmar purge and trap system for the HAFB investigation. The new electrically controlled injection valve system was found to be more rugged than the manual valve unit.
26. Sample pretreatment for semivolatile samples should be separated from the VOC sample analysis area to eliminate sample cross-contamination during the sample extraction process.
27. Good ventilation is critical to prevent sample cross-contamination.
28. All instruments can be electronically linked to a data management computer system for ease of data review and site map generation.

6.0 Summary of ETI Goals and Objectives

The funding provided by the U.S. Environmental Protection Agency as part of President Clinton's goal to strengthen the capacity of technology developers and users to succeed in environmental innovation enabled Tufts University and its manufacturing partners, Hewlett Packard, Bruker Analytical Instruments, Leeman Laboratories, TN Spectrace, CEM, and SiteWorks, to demonstrate their innovative field analytical instrumentation and methods during an ongoing site investigation at Hanscom Air Force Base. More than 800 analyses were made during the investigation for VOCs, PCBs, PAHs, and metals. EPA verified the quality of data produced in this project by conducting field and laboratory audits. The quality of field data produced in this project will allow its use in determining contaminant risk to ground water. The data produced in this investigation has resulted in design changes to the ground water collection system. These changes have already increased the concentration of contaminated ground water into the treatment facility.

The HAFB site investigation employed a dynamic on-site decision making framework. The core technical team included staff from EPA Region I, Massachusetts Department of Environmental Protection, Hanscom Air Force Base, CH2MHill, CDM, and Tufts. The technical team participated in the creation of the dynamic workplan and in carrying out the field investigation. This process, from planning to implementation, was videotaped and provided the basis for a dynamic workplan guideline produced by Tufts University in cooperation with EPA Region I, Office of Site Remediation and Restoration *and* Office of Environmental Measurement and Evaluation.

The guideline, videotape, and HAFB report should provide users of environmental data with confidence that screening and quantitative data can be produced in the field and that the data generated will be accepted by the regulatory community. The success at HAFB should reduce regulator uncertainty as to the quality of data that can be produced with these innovative technologies and their cost competitiveness. Without regulatory acceptance, consulting and remediation companies will continue to be reluctant to purchase field analytical services for site investigation and cleanup projects. Analytical service providers will not purchase new instrumentation or technologies or adopt innovative methods of analysis unless their customers

(environmental consulting and remediation companies as well as federal and state regulators) request their usage. This will result in continued reluctance by instrument manufacturers to develop, license, manufacture, or market innovative field analytical instrumentation.

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